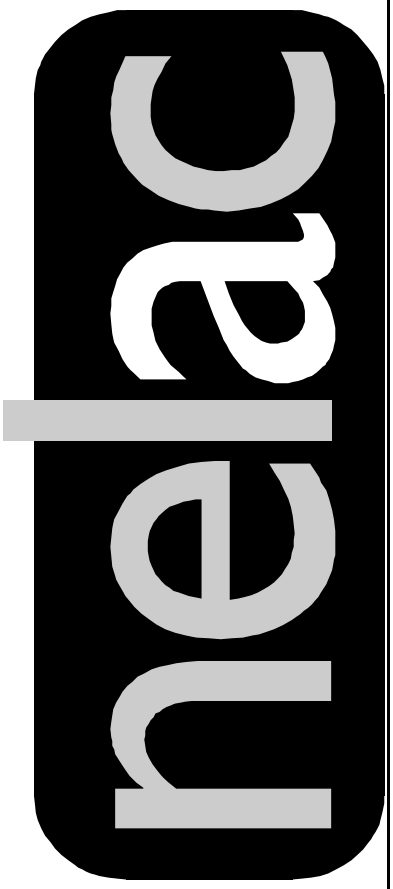
National Environmental Laboratory **Accreditation** Conference



QUALITY SYSTEMS

PROPOSED

June 2, 2003

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QUALITY SYSTEMS

NOTE: Additions (double-underlined) and ~~deletions~~ (struck through) to the approved standards being proposed for vote at the next Annual Meeting are marked as in this note.

5.0 QUALITY SYSTEMS

INTRODUCTION

~~Quality Systems include all quality assurance (QA) policies and quality control (QC) procedures, which shall be delineated in a Quality Manual and followed to ensure and document the quality of the analytical data. Laboratories seeking accreditation under NELAP must assure implementation of all QA policies and the essential applicable QC procedures specified in this Chapter. The QA policies, which establish essential QC procedures, are applicable to environmental laboratories regardless of size and complexity.~~

~~The intent of this Chapter is to provide sufficient detail concerning quality system requirements so that all accrediting authorities evaluate laboratories consistently and uniformly~~.

Each laboratory shall have a quality system. The laboratory's quality system is the process by which the laboratory conducts its activities so as to provide the client with data of known and documented quality with which to demonstrate regulatory compliance and for other decision-making purposes. This system includes a process by which appropriate analytical methods are selected, their capability is evaluated and their performance is documented. The quality system shall be documented in the laboratory's quality manual.

This chapter contains detailed quality system requirements for consistent and uniform implementation by both the laboratories conducting testing under these standards and the evaluation of those laboratories by accrediting authorities. Each laboratory seeking accreditation under NELAP must assure that they are implementing their quality system and that all Quality Control (QC) procedures specified in this chapter are being followed. The Quality Assurance (QA) policies, which establish QC procedure, are applicable to environmental laboratories regardless of size and complexity.

~~NELAC is committed to the use of Performance-based Measurement Systems (PBMS) in environmental testing and provides the foundation for PBMS implementation in these standards. While this standard may not currently satisfy all the anticipated needs of PBMS, NELAC will address future needs within the context of State statutory and regulatory requirements and the finalized EPA implementation plans for PBMS.~~

The growth in use of quality systems generally has increased the need to ensure that laboratories which form part of larger organizations or offer other services can operate to a quality system that is seen as compliant with ISO 9001 or ISO 9002 as well as with this Standard. Care has been taken, therefore, to incorporate all those requirements of ISO 9001 and ISO 9002 that are relevant to the scope of environmental testing ~~and calibration~~ services that are covered by the laboratory's quality system.

Environmental testing ~~and calibration~~ laboratories that comply with this Standard will therefore also operate in accordance with ISO 9001 or ISO 9002.

Certification against ISO 9001 and ISO 9002 does not of itself demonstrate the competence of the laboratory to produce technically valid data and results.

Chapter 5 is organized according to the structure of ISO/IEC 17025, 1999. Where deemed necessary, specific areas within this Chapter may contain more information than specified by ISO/IEC 17025.

All items identified in this Chapter shall be available for on-site inspection and data audit.

5.1 SCOPE

5.1.1 This Standard specifies the general requirements for the competence to carry out environmental tests ~~and/or calibrations~~, including sampling. It covers testing ~~and calibration~~ performed using standard methods, non-standard methods, and laboratory-developed methods.

It contains all of the requirements that environmental testing ~~and calibration~~ laboratories have to meet if they wish to demonstrate that they operate a quality system, are technically competent, and are able to generate technically valid results.

If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. (See the supplemental accreditation requirements in Section 1.8.2.)

5.1.2 This Standard is applicable to all organizations performing environmental tests ~~and/or calibrations~~. These include, for example, first-, second- and third-party laboratories, and laboratories where environmental testing ~~and/or calibration~~ forms part of inspection and product certification.

This Standard is applicable to all laboratories regardless of the number of personnel or the extent of the scope of environmental testing ~~and/or calibration~~ activities. When a laboratory does not undertake one or more of the activities covered by this Standard, such as sampling and the design/development of new methods, the requirements of those clauses do not apply.

5.1.3 The notes given provide clarification of the text, examples and guidance. They do not contain requirements and do not form an integral part of this Standard.

5.1.4 This Standard is for use by laboratories in developing their quality, administrative and technical systems that govern their operations. Laboratory clients, regulatory authorities and accreditation authorities may also use it in confirming or recognizing the competence of laboratories.

This Standard includes additional requirements and information for assessing competence or for determining compliance by the organization or accrediting authority granting the recognition (or approval).

5.1.5 Compliance with regulatory and safety requirements on the operation of laboratories is not covered by this Standard. It is the laboratory's responsibility to comply with the relevant health and safety requirements.

5.1.6 If environmental testing ~~and calibration~~ laboratories comply with the requirements of this Standard, they will operate a quality system for their environmental testing ~~and calibration~~ activities that also meets the requirements of ISO 9001 when they engage in the design/development of new methods, and/or develop test programs combining standard and non-standard test and calibration methods, and ISO 9002 when they only use standard methods. ISO/IEC 17025 covers several technical competence requirements that are not covered by ISO 9001 and ISO 9002.

5.1.7 An integral part of a Quality System is the data integrity procedures. The data integrity procedures provide assurance that a highly ethical approach to testing is a key component of all laboratory planning, training and implementation of methods. The following sections in this standard address data integrity procedures:

Management Responsibilities	5.4.2.6, 5.4.2.6.1, and 5.4.2.6.2
Training	5.5.2.7
Control and Documentation	5.4.15

5.2 REFERENCES

See Appendix A.

5.3 TERMS AND DEFINITIONS

The relevant definitions from ISO/IEC Guide 2, ANSI/ASQC E-4 (1994), and the International vocabulary of basic and general terms in metrology (VIM) are applicable, the most relevant being quoted in Appendix A, Glossary, of Chapter 1 together with further definitions applicable for the purposes of this Standard. General definitions related to quality are given in ISO 8402, whereas ISO/IEC Guide 2 gives definitions specifically related to standardization, certification, and laboratory accreditation. Where different definitions are given in ISO 8402, the definitions in ISO/IEC Guide 2 and VIM are preferred.

See Appendix A, Glossary, of Chapter 1.

5.4 MANAGEMENT REQUIREMENTS

5.4.1 Organization

5.4.1.1 The laboratory or the organization of which it is part shall be an entity that can be held legally responsible.

5.4.1.2 It is the responsibility of the laboratory to carry out its environmental testing ~~and calibration~~ activities in such a way as to meet the requirements of this Standard and to satisfy the needs of the client, the regulatory authorities or organizations providing recognition.

5.4.1.3 The laboratory management system shall cover work carried out in the laboratory's permanent facilities, at sites away from its permanent facilities, or in associated temporary or mobile facilities.

5.4.1.4 If the laboratory is part of an organization performing activities other than environmental testing ~~and/or calibration~~, the responsibilities of key personnel in the organization that have an involvement or influence on the environmental testing ~~and/or calibration~~ activities of the laboratory shall be defined in order to identify potential conflicts of interest.

a) Where a laboratory is part of a larger organization, the organizational arrangements shall be such that departments having conflicting interests, such as production, commercial marketing or financing do not adversely influence the laboratory's compliance with the requirements of this Standard.

b) The laboratory must be able to demonstrate that it is impartial and that it and its personnel are free from any undue commercial, financial and other pressures which might influence their technical judgment. Environmental ~~All environmental~~ testing ~~or calibration~~ laboratories

shall not engage in any activities that may endanger the trust in its independence of judgment and integrity in relation to its environmental testing ~~or calibration~~ activities.

5.4.1.5 The laboratory shall:

a) have managerial and technical personnel with the authority and resources needed to carry out their duties and to identify the occurrence of departures from the quality system or from the procedures for performing environmental tests ~~and/or calibrations~~, and to initiate actions to prevent or minimize such departures (see also 5.5.2);

b) have processes to ensure that its management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work;

c) have policies and procedures to ensure the protection of its clients' confidential information and proprietary rights, including procedures for protecting the electronic storage and transmission of results.

The policy and procedures to ensure the protection of clients' confidential information and proprietary rights may not apply to in-house laboratories.

d) have policies and procedures to avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgment or operational integrity;

e) define the organization and management structure of the laboratory, its place in any parent organization, and the relationships between quality management, technical operations and support services;

f) specify the responsibility, authority and interrelationships of all personnel who manage, perform or verify work affecting the quality of the environmental tests ~~and/or calibrations~~.

Documentation shall include a clear description of the lines of responsibility in the laboratory and shall be proportioned such that adequate supervision is ensured;

g) provide adequate supervision of environmental testing ~~and calibration~~ staff, including trainees, by persons familiar with methods and procedures, purpose of each environmental test ~~and/or calibration~~, and with the assessment of the environmental test ~~or calibration~~ results;

h) have technical management which has overall responsibility for the technical operations and the provision of the resources needed to ensure the required quality of laboratory operations;

The technical director(s) (however named) shall certify that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. Such certification shall be documented.

The technical director(s) shall meet the requirements specified in the Accreditation Process. (see 4.1.1.1)

i) appoint a member of staff as quality manager (however named) who, irrespective of other duties and responsibilities, shall have defined responsibility and authority for ensuring that the quality system is implemented and followed at all times; the quality manager shall have direct

access to the highest level of management at which decisions are made on laboratory policy or resources;

Where staffing is limited, the quality manager may also be the technical director or deputy technical director;

The quality manager (and/or his/her designees) shall:

1) serve as the focal point for QA/QC and be responsible for the oversight and/or review of quality control data;

2) have functions independent from laboratory operations for which they have quality assurance oversight;

3) be able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence;

4) have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system as defined under NELAC;

5) have a general knowledge of the analytical test methods for which data review is performed;

6) arrange for or conduct internal audits as per 5.4.13 annually; and,

7) notify laboratory management of deficiencies in the quality system and monitor corrective action.

j) appoint deputies for key managerial personnel. Including the technical director(s) and/or quality manager;

k) for purposes of qualifying for and maintaining accreditation, each laboratory shall participate in a proficiency test program as outlined in Chapter 2.

5.4.2 Quality System

5.4.2.1 The laboratory shall establish implement and maintain a quality system based on the required elements contained in this chapter and appropriate to the type, range and volume of environmental testing activities it undertakes. The laboratory shall document its policies, systems, programs, procedures and instructions to the extent necessary to assure the quality of the environmental test ~~and/or calibration~~ results. The system's documentation shall be communicated to, understood by, available to, and implemented by the appropriate personnel.

5.4.2.2 The laboratory's quality system policies and objectives shall be defined in a quality manual (however named). The overall objectives shall be documented in a quality policy statement. The quality policy statement shall be issued under the authority of the chief executive. It shall include at least the following:

a) the laboratory management's commitment to good professional practice and to the quality of its environmental testing ~~and calibration~~ in servicing its clients; The laboratory shall define and document its policies and objectives for, and its commitment to accepted laboratory practices and quality of testing services.

b) the management's statement of the laboratory's standard of service;

c) the objectives of the quality system;

The laboratory management shall ensure that these policies and objectives are documented in a quality manual.

d) a requirement that all personnel concerned with environmental testing ~~and calibration~~ activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work; and

e) the laboratory management's commitment to compliance with this Standard.

5.4.2.3 The quality manual shall include or make reference to the supporting procedures including technical procedures. It shall outline the structure of the documentation used in the quality system.

The quality manual, and related quality documentation, shall state the laboratory's policies and operational procedures established in order to meet the requirements of this Standard.

Where a laboratory's quality manual contains the necessary requirements, a separate SOP or policy is not required.

The quality manual shall list on the title page: a document title; the laboratory's full name and address; the name, address (if different from above), and telephone number of individual(s) responsible for the laboratory; the name of the quality manager (however named); the identification of all major organizational units which are to be covered by this quality manual and the effective date of the version;

The quality manual and related quality documentation shall also contain:

a) a quality policy statement, including objectives and commitments, by top management (see 5.4.2.2);

b) the organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;

c) the relationship between management, technical operations, support services and the quality system;

d) procedures to ensure that all records required under this Chapter are retained, as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures (SOPs), manuals, or documents clearly indicate the time period during which the procedure or document was in force;

e) job descriptions of key staff and reference to the job descriptions of other staff;

f) identification of the laboratory's approved signatories; at a minimum, the title page of the Quality Manual must have the signed and dated concurrence, (with appropriate titles) of all responsible parties including the quality manager(s), technical director(s), and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager;

g) the laboratory's procedures for achieving traceability of measurements;

h) a list of all test methods under which the laboratory performs its accredited testing;

i) mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;

j) reference to the calibration and/or verification test procedures used;

k) procedures for handling submitted samples;

l) reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests;

m) reference to procedures for calibration, verification and maintenance of equipment;

n) reference to verification practices which may include interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;

o) procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;

p) the laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;

q) procedures for dealing with complaints;

r) procedures for protecting confidentiality (including national security concerns), and proprietary rights;

s) procedures for audits and data review;

t) processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training;

u) reference to procedures for reporting analytical results; and,

v) a Table of Contents, and applicable lists of references and glossaries, and appendices.

5.4.2.4 The roles and responsibilities of technical management and the quality manager, including their responsibility for ensuring compliance with this Standard, shall be defined in the quality manual.

5.4.2.5 The quality manual shall be maintained current under the responsibility of the quality manager.

5.4.2.6 The laboratory shall establish and maintain data integrity procedures. These procedures shall be defined in detail within the quality manual. There are four required elements within a data integrity system. These are 1) data Integrity training, 2) signed data integrity documentation for all laboratory employees, 3) in-depth, periodic monitoring of data integrity, and 4) data integrity procedure documentation. The data integrity ~~integrity~~ procedures shall be signed and dated by senior management. These procedures and the associated implementation records shall be properly maintained and made available for assessor review. The data integrity procedures shall be annually reviewed and updated by management.

5.4.2.6.1 Laboratory management shall provide a mechanism for confidential reporting of data integrity issues in their laboratory. A primary element of the mechanism is to assure confidentiality and a receptive environment in which all employees may privately discuss ethical issues or report items of ethical concern.

5.4.2.6.2 In instances of ethical concern, the mechanism shall include a process whereby laboratory ~~Laboratory~~ management are to be informed of the need for any further detailed investigation.

5.4.3 Document Control

5.4.3.1 General

The laboratory shall establish and maintain procedures to control all documents that form part of its quality system (internally generated or from external sources), ~~such as regulations, standards, other normative documents, environmental test and/or calibration methods, as well as drawings, software, specifications, instructions and manuals.~~ Documents include policy statements, procedures, specifications, calibration tables, charts, textbooks, posters, notices, memoranda, software, drawings, plans, etc. These may be on various media, whether hard copy or electronic, and they may be digital, analog, photographic or written.

The control of data related to environmental testing ~~and calibration~~ is covered in 5.5.4.7. The control of records is covered in 5.4.12.

5.4.3.2 Document Approval and Issue

5.4.3.2.1 All documents issued to personnel in the laboratory as part of the quality system shall be reviewed and approved for use by authorized personnel prior to issue. A master list or an equivalent document control procedure identifying the current revision status and distribution of documents in the quality system shall be established and be readily available to preclude the use of invalid and/or obsolete documents.

5.4.3.2.2 The procedure(s) adopted shall ensure that:

a) authorized editions of appropriate documents are available at all locations where operations essential to the effective functioning of the laboratory are performed;

b) documents are periodically reviewed and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements;

c) invalid or obsolete documents are promptly removed from all points of issue or use, or otherwise assured against unintended use;

d) obsolete documents retained for either legal or knowledge preservation purposes are suitably marked.

5.4.3.2.3 Quality system documents generated by the laboratory shall be uniquely identified. Such identification shall include the date of issue and/or revision identification, page numbering, the total number of pages or a mark to signify the end of the document, and the issuing authority(ies).

5.4.3.3 Document Changes

5.4.3.3.1 Changes to documents shall be reviewed and approved by the same function that performed the original review unless specifically designated otherwise. The designated personnel shall have access to pertinent background information upon which to base their review and approval.

5.4.3.3.2 Where practicable, the altered or new text shall be identified in the document or the appropriate attachments.

5.4.3.3.3 If the laboratory's documentation control system allows for the amendment of documents by hand, pending the re-issue of the documents, the procedures and authorities for such amendments shall be defined. Amendments shall be clearly marked, initialed and dated. A revised document shall be formally re-issued as soon as practicable.

5.4.3.3.4 Procedures shall be established to describe how changes in documents maintained in computerized systems are made and controlled.

5.4.4 Review of Requests, Tenders and Contracts

5.4.4.1 The laboratory shall establish and maintain procedures for the review of requests, tenders and contracts. The policies and procedures for these reviews leading to a contract for environmental testing ~~and/or calibration~~ shall ensure that:

a) the requirements, including the methods to be used, are adequately defined, documented and understood (see 5.5.4.2);

b) the laboratory has the capability and resources to meet the requirements;

The purpose of this review of capability is to establish that the laboratory possesses the necessary physical, personnel and information resources, and that the laboratory's personnel have the skills and expertise necessary for the performance of the environmental tests ~~and/or calibrations~~ in question. The review may encompass results of earlier participation in interlaboratory comparisons or proficiency testing and/or the running of trial environmental test ~~or calibration~~ programs using samples or items of known value in order to determine uncertainties of measurement, detection limits, ~~of~~ confidence limits, or other essential quality control requirements. The current accreditation status of the laboratory must also be reviewed. The laboratory must inform the client of the results of this review if it indicates any potential conflict, deficiency, lack of appropriate accredtation status, or inability on the laboratory's part to complete the client's work.

c) the appropriate environmental test ~~and/or calibration~~ method is selected and capable of meeting the clients' requirements (see 5.5.4.2).

Any differences between the request or tender and the contract shall be resolved before any work commences. Each contract shall be acceptable both to the laboratory and the client.

A contract may be any written or oral agreement to provide a client with environmental testing ~~and/or calibration~~ services.

5.4.4.2 Records of reviews, including any significant changes, shall be maintained. Records shall also be maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract.

For review of routine and other simple tasks, the date and the identification (e. g. the initials) of the person in the laboratory responsible for carrying out the contracted work are considered adequate.

For repetitive routine tasks, the review need be made only at the initial inquiry ~~enquiry~~ stage or on granting of the contract for on-going routine work performed under a general agreement with the client, provided that the client's requirements remain unchanged. For new, complex or advanced environmental testing ~~and/or calibration~~ tasks, a more comprehensive record should be maintained.

5.4.4.3 The review shall also cover any work that is subcontracted by the laboratory.

5.4.4.4 The client shall be informed of any deviation from the contract.

5.4.4.5 If a contract needs to be amended after work has commenced, the same contract review process shall be repeated and any amendments shall be communicated to all affected personnel. Suspension of accreditation, revocation of accreditation, or voluntary withdrawal of accreditation must be reported to the client.

5.4.5 Subcontracting of Environmental Tests ~~and Calibrations~~

5.4.5.1 When a laboratory subcontracts work whether because of unforeseen reasons (e. g. workload, need for further expertise or temporary incapacity) or on a continuing basis (e. g. through permanent subcontracting, agency or franchising arrangements), this work shall be placed with a laboratory accredited under NELAP for the tests to be performed or with a laboratory that meets applicable statutory and regulatory requirements for performing the tests and submitting the results of tests performed. The laboratory performing the subcontracted work shall be indicated in the final report and non-NELAP accredited work shall be clearly identified.

5.4.5.2 The laboratory shall advise the client of the arrangement in writing and, when possible ~~appropriate~~, gain the approval of the client, preferably in writing.

5.4.5.3 The laboratory is responsible to the client for the subcontractor's work, except in the case where the client or a regulatory authority specifies which subcontractor is to be used.

5.4.5.4 The laboratory shall maintain a register of all subcontractors that it uses for environmental tests ~~and/or calibrations~~ and a record of the evidence of compliance with 5.4.5.1.

5.4.6 Purchasing Services and Supplies

5.4.6.1 The laboratory shall have a policy and procedure(s) for the selection and purchasing of services and supplies it uses that affect the quality of the environmental tests ~~and/or calibrations~~. Procedures shall exist for the purchase, reception and storage of reagents and laboratory consumable materials relevant for the environmental tests ~~and calibrations~~.

5.4.6.2 The laboratory shall ensure that purchased supplies and reagents and consumable materials that affect the quality of environmental tests ~~and/or calibrations~~ are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements defined in the methods for the environmental tests ~~and/or calibrations~~ concerned. These services and supplies used shall comply with specified requirements. Records of actions taken to check compliance shall be maintained.

5.4.6.3 Purchasing documents for items affecting the quality of laboratory output shall contain data describing the services and supplies ordered. These purchasing documents shall be reviewed and approved for technical content prior to release.

5.4.6.4 The laboratory shall evaluate suppliers of critical consumables, supplies and services which affect the quality of environmental testing ~~and calibration~~, and shall maintain records of these evaluations and list those approved.

5.4.7 Service to the Client

The laboratory shall afford clients or their representatives cooperation to clarify the client's request and to monitor the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other clients.

5.4.8 Complaints

The laboratory shall have a policy and procedure for the resolution of complaints received from clients or other parties. Records shall be maintained of all complaints and of the investigations and corrective actions taken by the laboratory (see also 5.4.10).

5.4.9 Control of Nonconforming Environmental Testing ~~and/or Calibration~~ Work

5.4.9.1 The laboratory shall have a policy and procedures that shall be implemented when any aspect of its environmental testing ~~and/or calibration~~ work, or the results of this work, do not conform to its own procedures or the agreed requirements of the client. The policy and procedures shall ensure that:

a) the responsibilities and authorities for the management of nonconforming work are designated and actions (including halting of work and withholding of test reports ~~and calibration certificates~~, as necessary) are defined and taken when nonconforming work is identified;

b) an evaluation of the significance of the nonconforming work is made;

c) corrective actions are taken immediately, together with any decision about the acceptability of the nonconforming work;

d) where the data quality is or may be impacted ~~necessary~~, the client is notified and work may be ~~is~~ recalled;

e) the responsibility for authorizing the resumption of work is defined.

5.4.9.2 Where the evaluation indicates that the nonconforming work could recur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures, the corrective action procedures given in 5.4.10 shall be promptly followed.

5.4.10 Corrective Action

5.4.10.1 General

The laboratory shall establish a policy and procedure and shall designate appropriate authorities for implementing corrective action when nonconforming work or departures from the policies and procedures in the quality system or technical operations have been identified.

5.4.10.2 Cause Analysis

The procedure for corrective action shall start with an investigation to determine the root cause(s) of the problem.

5.4.10.3 Selection and Implementation of Corrective Actions

Where corrective action is needed, the laboratory shall identify potential corrective actions. It shall select and implement the action(s) most likely to eliminate the problem and to prevent recurrence.

Corrective actions shall be to a degree appropriate to the magnitude and the risk of the problem.

The laboratory shall document and implement any required changes resulting from corrective action investigations.

5.4.10.4 Monitoring of Corrective Actions

The laboratory shall monitor the results to ensure that the corrective actions taken have been effective.

5.4.10.5 Additional Audits

Where the identification of nonconformances or departures casts doubts on the laboratory's compliance with its own policies and procedures, or on its compliance with this Standard, the laboratory shall ensure that the appropriate areas of activity are audited in accordance with 5.4.13 as soon as possible.

5.4.10.6 Technical Corrective Action

a) In addition to providing acceptance criteria and specific protocols for corrective actions in the Method SOPs (see 5.5.4.1.1), the laboratory shall implement general procedures to be followed to determine when departures from documented policies, procedures and quality control have occurred. These procedures shall include but are not limited to the following:

   1) identify the individual(s) responsible for assessing each QC data type;

   2) identify the individual(s) responsible for initiating and/or recommending corrective actions;

   3) define how the analyst shall treat a data set if the associated QC measurements are unacceptable;

   4) specify how out-of-control situations and subsequent corrective actions are to be documented; and,

   5) specify procedures for management (including the quality manager) to review corrective action reports.

b) To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate laboratory defined data qualifier(s).

5.4.11 Preventive Action

Preventive action is a pro-active process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

5.4.11.1 Needed improvements and potential sources of nonconformances, either technical or concerning the quality system, shall be identified. If preventive action is required, action plans shall be developed, implemented and monitored to reduce the likelihood of the occurrence of such nonconformances and to take advantage of the opportunities for improvement.

5.4.11.2 Procedures for preventive actions shall include the initiation of such actions and application of controls to ensure that they are effective.

5.4.12 Control of Records

The laboratory shall maintain a record system to suit its particular circumstances and comply with any applicable regulations. The system shall produce unequivocal, accurate records which document all laboratory activities. The laboratory shall retain all original observations, calculations and derived data, calibration records and a copy of the test report for a minimum of five years.

There are two levels of sample handling: 1) sample tracking and 2) legal chain of custody protocols, which are used for evidentiary or legal purposes. All essential requirements for sample tracking (e. g., chain of custody form) are outlined in Sections 5.4.12.1.5, 5.4.12.2.4 and 5.4.12.2.5. If a client specifies that a sample will be used for evidentiary purposes, then a laboratory shall have a written SOP for how that laboratory will carry out legal chain of custody for example, ASTM D 4840- 95 and Manual for the Certification of Laboratories Analyzing Drinking Water, March 1997, Appendix A.

5.4.12.1 General

5.4.12.1.1 The laboratory shall establish and maintain procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. Quality records shall include reports from internal audits and management reviews as well as records of corrective and preventive actions. Records may be in any media, such as hard copy or electronic media.

5.4.12.1.2 All records shall be legible and shall be stored and retained in such a way that they are readily retrievable in facilities that provide a suitable environment to prevent damage or deterioration and to prevent loss. Retention times of records shall be established.

5.4.12.1.3 All records shall be held secure and in confidence.

5.4.12.1.4 The laboratory shall have procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records.

5.4.12.1.5 The record keeping system must allow historical reconstruction of all laboratory activities that produced the analytical data. The history of the sample must be readily understood through the documentation. This shall include interlaboratory transfers of samples and/or extracts.

a) The records shall include the identity of personnel involved in sampling, sample receipt, preparation~~, calibration~~ or testing.

b) All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification shall be documented.

c) The record keeping system shall facilitate the retrieval of all working files and archived records for inspection and verification purposes, e.g., set format for naming electronic files.

d) All changes to records shall be signed or initialed by responsible staff. The reason for the signature or initials shall be clearly indicated in the records such as "sampled by," "prepared by," or "reviewed by."

e) All generated data except those that are generated by automated data collection systems, shall be recorded directly, promptly and legibly in permanent ink.

f) Entries in records shall not be obliterated by methods such as erasures, overwritten files or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. These criteria also shall apply to electronically maintained records.

g) Refer to 5.5.4.7.2 for Computer and Electronic Data.

5.4.12.2 Technical Records

5.4.12.2.1 The laboratory shall retain records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each test report ~~or calibration certificate~~ issued, for a defined period. The records for each environmental test ~~or calibration~~ shall contain sufficient information to facilitate~~, if possible,~~ identification of factors affecting the uncertainty and to enable the environmental test ~~or calibration~~ to be repeated under conditions as close as possible to the original. The records shall include the identity of personnel responsible for the sampling, performance of each environmental test ~~and/or calibration~~ and checking of results.

5.4.12.2.2 Observations, data and calculations shall be recorded at the time they are made and shall be identifiable to the specific task.

5.4.12.2.3 When mistakes occur in records, each mistake shall be crossed out, not erased, made illegible or deleted, and the correct value entered alongside. All such alterations to records shall be signed or initialed by the person making the correction. In the case of records stored electronically, equivalent measures shall be taken to avoid loss or change of original data.

When corrections are due to reasons other than transcription errors, the reason for the correction shall be documented.

5.4.12.2.4 Records Management and Storage

a) All records (including those pertaining to ~~calibration and~~ test equipment), certificates and reports shall be safely stored, held secure and in confidence to the client. NELAP-related records shall be available to the accrediting authority.

b) All records, including those specified in 5.4.12.2.5 shall be retained for a minimum of five years from generation of the last entry in the records. All information necessary for the historical reconstruction of data must be maintained by the laboratory. Records which are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

c) Records that are stored or generated by computers or personal computers shall have hard copy or write-protected backup copies.

d) The laboratory shall establish a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting.

e) Access to archived information shall be documented with an access log. These records shall be protected against fire, theft, loss, environmental deterioration, vermin and, in the case of electronic records, electronic or magnetic sources.

f) The laboratory shall have a plan to ensure that the records are maintained or transferred according to the clients' instructions (see 4.1.8.e) in the event that a laboratory transfers ownership or goes out of business. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed.

5.4.12.2.5 Laboratory Sample Tracking

5.4.12.2.5.1 Sample Handling

A record of all procedures to which a sample is subjected while in the possession of the laboratory shall be maintained. These shall include but are not limited to all records pertaining to:

a) sample preservation including appropriateness of sample container and compliance with holding time requirement;

b) sample identification, receipt, acceptance or rejection and log-in;

c) sample storage and tracking including shipping receipts, sample transmittal forms, (chain of custody form); and

d) ~~the laboratory shall have~~ documented procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

5.4.12.2.5.2 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following shall be retained:

a) all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);

b) a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;

c) copies of final reports;

d) archived SOPs;

e) correspondence relating to laboratory activities for a specific project;

f) all corrective action reports, audits and audit responses;

g) proficiency test results and raw data; and,

h) results of data review, verification, and cross-checking procedures.

5.4.12.2.5.3 Analytical Records

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, shall include:

a) laboratory sample ID code;

b) date of analysis and time of analysis is required if the holding time is 72 hours or less or when time critical steps are included in the analysis, e.g., extractions, and incubations;

c) instrumentation identification and instrument operating conditions/parameters (or reference to such data);

d) analysis type;

e) all manual calculations, e.g., manual integrations; and,

f) analyst's or operator's initials/signature;

g) sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

h) sample analysis;

i) standard and reagent origin, receipt, preparation, and use;

j) calibration criteria, frequency and acceptance criteria;

k) data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

l) quality control protocols and assessment;

m) electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

n) method performance criteria including expected quality control requirements.

5.4.12.2.5.4 Administrative Records

The following shall be maintained:

a) personnel qualifications, experience and training records;

b) records of demonstration of capability for each analyst; and

c) a log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.

5.4.13 Internal Audits

5.4.13.1 The laboratory shall periodically, in accordance with a predetermined schedule and procedure, and at least annually, conduct internal audits of its activities to verify that its operations continue to comply with the requirements of the quality system and this Standard. The internal audit program shall address all elements of the quality system, including the environmental testing ~~and/or calibration~~ activities. It is the responsibility of the quality manager to plan and organize audits as required by the schedule and requested by management. Such audits shall be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited. Personnel shall not audit their own activities except when it can be demonstrated that an effective audit will be carried out.

5.4.13.2 When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's environmental test ~~or calibration~~ results, the laboratory shall take timely corrective action, and shall notify clients in writing if investigations show that the laboratory results may have been affected.

The laboratory shall notify clients promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any ~~calibration certificate,~~ test report or test certificate or amendment to a report or certificate.

<u>The laboratory must specify, in the laboratory's quality manual, the time frame for notifying a client of events that cast doubt on the validity results.</u>

5.4.13.3 The area of activity audited, the audit findings and corrective actions that arise from them shall be recorded. The laboratory management shall ensure that these actions are discharged within the agreed time frame as indicated in the quality manual and/or SOPs.

5.4.13.4 Follow-up audit activities shall verify and record the implementation and effectiveness of the corrective action taken.

5.4.14 Management Reviews

5.4.14.1 In accordance with a predetermined schedule and procedure, the laboratory's executive management shall periodically and at least annually conduct a review of the laboratory's quality system and environmental testing ~~and/or calibration~~ activities to ensure their continuing suitability and effectiveness, and to introduce necessary changes or improvements. The review shall take account of:

—a) the suitability of policies and procedures;

—b) reports from managerial and supervisory personnel;

—c) the outcome of recent internal audits;

—d) corrective and preventive actions;

—e) assessments by external bodies;

—f) the results of interlaboratory comparisons or proficiency tests;

—g) changes in the volume and type of the work;

—h) client feedback;

—i) complaints;

—j) other relevant factors, such as quality control activities, resources and staff training.

5.4.14.2 Findings from management reviews and the actions that arise from them shall be recorded. The management shall ensure that those actions are carried out within an appropriate and agreed timescale.

The laboratory shall have a procedure for review by management and maintain records of review findings and actions.

5.4.15 The laboratory, as part of their overall internal auditing program, shall insure that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery of potential issues shall be handled in a confidential manner until such time as a follow up evaluation, full investigation, or other appropriate actions have been completed and the issues clarified. All investigations that result in finding of inappropriate activity shall be documented and shall include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients. All documentation of these investigation and actions taken shall be maintained for at least five years.

5.5 TECHNICAL REQUIREMENTS

5.5.1 General

5.5.1.1 Many factors determine the correctness and reliability of the environmental tests ~~and/or calibrations~~ performed by a laboratory. These factors include contributions from:

a) human factors (5.5.2);

b) accommodation and environmental conditions (5.5.3);

c) environmental test ~~and calibration~~ methods and method validation (5.5.4);

d) equipment (5.5.5);

e) measurement traceability (5.5.6);

f) sampling (5.5.7);

g) the handling of samples (5.5.8).

5.5.1.2 The extent to which the factors contribute to the total uncertainty of measurement differs considerably between (types of) environmental tests ~~and between (types of) calibrations~~. The laboratory shall take account of these factors in developing environmental test ~~and calibration~~ methods and procedures, in the training and qualification of personnel, and in the selection and calibration of the equipment it uses.

5.5.2 Personnel

5.5.2.1 The laboratory management shall ensure the competence of all who operate specific equipment, perform environmental tests ~~and/or calibrations~~, evaluate results, and sign test reports ~~and calibration certificates~~. When using staff who are undergoing training, appropriate supervision shall be provided. Personnel performing specific tasks shall be qualified on the basis of appropriate education, training, experience and/or demonstrated skills, as required.

The laboratory shall have sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned functions.

All personnel shall be responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function. Each technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular function and a general knowledge of laboratory operations, test methods, quality assurance/quality control procedures and records management.

5.5.2.2 The management of the laboratory shall formulate the goals with respect to the education, training and skills of the laboratory personnel. The laboratory shall have a policy and procedures for identifying training needs and providing training of personnel. The training program shall be relevant to the present and anticipated tasks of the laboratory.

5.5.2.3 The laboratory shall use personnel who are employed by, or under contract to, the laboratory. Where contracted and additional technical and key support personnel are used, the laboratory shall ensure that such personnel are supervised and competent and that they work in accordance with the laboratory's quality system.

5.5.2.4 The laboratory shall maintain current job descriptions for all personnel who manage, perform, or verify work affecting the quality of the environmental tests ~~and/or calibrations.~~

5.5.2.5 The management shall authorize specific personnel to perform particular types of sampling, environmental <u>testing</u> ~~test and/or calibration~~, to issue test reports ~~and calibration certificates~~, to give opinions and interpretations and to operate particular types of equipment. The laboratory shall maintain records of the relevant authorization(s), competence, educational and professional qualifications, training, skills and experience of all technical personnel, including contracted personnel. This information shall be readily available and shall include the date on which authorization and/or competence is confirmed.

Records on the relevant qualifications, training, skills and experience of the technical personnel shall be maintained by the laboratory [see 5.5.2.6.c], including records on demonstrated proficiency for each laboratory test method, such as the criteria outlined in 5.5.4.2.2 for chemical testing.

5.5.2.6 The laboratory management shall be responsible for:

a) defining the minimal level of qualification, experience and skills necessary for all positions in the laboratory. In addition to education and/or experience, basic laboratory skills such as using a balance, colony counting, aseptic or quantitative techniques shall be considered;

b) ensuring that all technical laboratory staff have demonstrated capability in the activities for which they are responsible. Such demonstration shall be documented. (See Appendix C);

Note: In laboratories with specialized “work cells” (a well defined group of analysts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented.

c) ensuring that the training of each member of the technical staff is kept up-to-date (on-going) by the following:

1) Evidence must be on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house quality documentation, which relates to his/her job responsibilities.

2) Training courses or workshops on specific equipment, analytical techniques or laboratory procedures shall all be documented.

3) Analyst training shall be considered up to date if an employee training file contains a certification that technical personnel have read, understood and agreed to perform the most recent version of the test method (the approved method or standard operating procedure as defined by the laboratory document control system, 5.4.2.3.d) and documentation of continued proficiency by at least one of the following once per year:

i. acceptable performance of a blind sample (single blind to the analyst). Note: ~~;~~

~~ii. another demonstration of capability;~~

~~iii.~~ successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624 or 5030 ~~5035~~/8260) would only require documentation for one of the test methods. The laboratory must determine the acceptable range of the blind performance sample prior to analysis;

ii. an initial measurement system evaluation or another demonstration of capability;

iii~~iv~~. at least four consecutive laboratory control samples with acceptable levels of precision and accuracy. The laboratory must determine the acceptable range for precision and accuracy prior to analysis; or

iv. if i-iii ~~i-iv~~ cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.

d) documenting all analytical and operational activities of the laboratory;

e) supervising all personnel employed by the laboratory.

f) ensuring that all sample acceptance criteria (Section 5.5.8) are verified and that samples are logged into the sample tracking system and properly labeled and stored;

g) documenting the quality of all data reported by the laboratory; and

5.5.2.7 Data integrity training shall be provided as a formal part of new employee orientation and must also be provided on an annual basis for all current employees. Topics covered shall be documented in writing and provided to all trainees. Key topics covered during training must include organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, how and when to report data integrity issues, and record keeping. Training shall include discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedure documentation. Employees are required to understand that any infractions of the laboratory data integrity procedures will result in a detailed

investigation that could lead to very serious consequences including immediate termination, debarment or civil/criminal prosecution. The initial data integrity training and the annual refresher training shall have a signature attendance sheet or other form of documentation that demonstrates all staff have participated and understand their obligations related to data integrity. Senior managers acknowledge their support of these procedures by 1) upholding the spirit and intent of the organization's data integrity procedures and 2) effectively implementing the specific requirements of the procedures.

Specific examples of breaches of ethical behavior should be discussed including improper data manipulations, adjustments of instrument time clocks, and inappropriate changes in concentrations of standards. Data integrity training requires emphasis on the importance of proper written narration on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially deficient. The data integrity procedures may also include written ethics agreements, examples of improper practices, examples of improper chromatographic manipulations, requirements for external ethics program training, and any external resources available to employees.

5.5.3 Accommodation and Environmental Conditions

5.5.3.1 Laboratory facilities for environmental testing ~~and/or calibration~~, including but not limited to energy sources, lighting and environmental conditions, shall be such as to facilitate correct performance of the environmental tests ~~and/or calibrations~~.

The laboratory shall ensure that the environmental conditions do not invalidate the results or adversely affect the required quality of any measurement. Particular care shall be taken when sampling and environmental tests ~~and/or calibrations~~ are undertaken at sites other than a permanent laboratory facility. The technical requirements for accommodation and environmental conditions that can affect the results of environmental tests ~~and calibrations~~ shall be documented.

5.5.3.2 The laboratory shall monitor, control and record environmental conditions as required by the relevant specifications, methods and procedures or where they influence the quality of the results. Due attention shall be paid, for example, to biological sterility, dust, electromagnetic disturbances, radiation, humidity, electrical supply, temperature, and sound and vibration levels, as appropriate to the technical activities concerned. Environmental tests ~~and calibrations~~ shall be stopped when the environmental conditions jeopardize the results of the environmental tests ~~and/or calibrations~~.

In instances where monitoring or control of any of the above mentioned items are specified in a test method or by regulation, the laboratory shall meet and document adherence to the laboratory facility requirements.

5.5.3.3 There shall be effective separation between neighboring areas in which there are incompatible activities including culture handling or incubation areas and volatile organic chemicals handling areas. Measures shall be taken to prevent cross-contamination.

5.5.3.4 Access to and use of areas affecting the quality of the environmental tests ~~and/or calibrations~~ shall be controlled. The laboratory shall determine the extent of control based on its particular circumstances.

5.5.3.5 Measures shall be taken to ensure good housekeeping in the laboratory. Special procedures shall be prepared where necessary.

5.5.3.6 Work spaces must be available to ensure an unencumbered work area. Work areas include:

a) access and entryways to the laboratory;

b) sample receipt area(s);

c) sample storage area(s);

d) chemical and waste storage area(s); and,

e) data handling and storage area(s).

5.5.4 Environmental Test ~~and Calibration~~ Methods and Method Validation

5.5.4.1 General

The laboratory shall use appropriate methods and procedures for all environmental tests ~~and/or calibrations~~ within its scope. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement uncertainty as well as statistical techniques for analysis of environmental test ~~and/or calibration~~ data.

The laboratory shall have instructions on the use and operation of all relevant equipment, and on the handling and preparation of samples where the absence of such instructions could jeopardize the results of environmental tests ~~and/or calibrations~~. All instructions, standards, manuals and reference data relevant to the work of the laboratory shall be kept up to date and shall be made readily available to personnel (see 5.4.3). Deviation from environmental test ~~and calibration~~ methods shall occur only if the deviation has been documented, technically justified, authorized, and accepted by the client.

5.5.4.1.1 Standard Operating Procedures (SOPs)

Laboratories shall maintain SOPs that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods.

a) These documents, for example, may be equipment manuals provided by the manufacturer, or internally written documents with adequate detail to allow someone similarly qualified, other than the analyst, to reproduce the procedures used to generate the test result.

b) The test methods may be copies of published methods as long as any changes or selected options in the methods are documented and included in the methods manual (see 5.5.4.1.2).

c) Copies of all SOPs shall be accessible to all personnel.

d) The SOPs shall be organized.

e) Each SOP shall clearly indicate the effective date of the document, the revision number and the signature(s) of the approving authority.

f) The documents specified in 5.5.4.1.1 a) and 5.5.4.1.1 b) that contain sufficient information to perform the tests do not need to be supplemented or rewritten as internal procedures, if the documents are written in a way that they can be used as written. Any changes, including the use of a selected option must be documented and included in the laboratory's methods manual.

5.5.4.1.2 Laboratory Method Manual(s)

a) The laboratory shall have and maintain an in-house methods manual(s) for each accredited analyte or test method.

b) This manual may consist of copies of published or referenced test methods or SOPs that have been written by the laboratory. In cases where modifications to the published method have been made by the laboratory or where the referenced test method is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described. Each test method shall include or reference where applicable:

1) identification of the test method;
~~2) applicable matrix or matrices;~~
~~3) detection limit;~~
~~4)~~2) Scope and application, including components to be analyzed;
~~5)~~3) summary of the test method;
~~6)~~4) definitions;
~~7)~~5) interferences;
~~8)~~6) safety;
~~9)~~7) equipment and supplies;
~~10)~~8) reagents and standards;
~~11)~~9) sample collection, preservation, shipment and storage;
~~12)~~10) quality control;
~~13)~~11) calibration and standardization;
~~14)~~12) procedure;
~~15)~~13) data analysis and calculations;
~~16)~~14) method performance;
~~17)~~15) pollution prevention;
~~18) data assessment and acceptance criteria for quality control measures;~~
~~19) corrective actions for out-of-control data;~~
~~20) contingencies for handling out-of-control or unacceptable data;~~
~~21)~~16) waste management;
~~22)~~17) references; and,
~~23)~~18) any tables, diagrams, flowcharts and validation data.

5.5.4.2 Selection of Methods

The laboratory shall use methods for environmental testing ~~and/or calibration~~, including methods for sampling, which meet the needs of the client and which are appropriate for the environmental tests ~~and/or calibrations~~ it undertakes.

5.5.4.2.1 Sources of Methods

a) Methods published in international, regional or national standards shall preferably be used. The laboratory shall ensure that it uses the latest valid edition of a standard unless it is not appropriate or possible to do so. When necessary, the standard shall be supplemented with additional details to ensure consistent application.

b) When the use of specific methods for a sample analysis are mandated or requested, only those methods shall be used.

c) When the client does not specify the method to be used or where methods are employed that are not required, ~~as in the Performance Based Measurement System approach,~~ the methods shall be fully documented and validated (see 5.5.4.2.2, 5.5.4.5, and Appendix C), and be

available to the client and other recipients of the relevant reports. The laboratory shall select appropriate methods that have been published either in international, regional or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment. Laboratory-developed methods or methods adopted by the laboratory may also be used if they are appropriate for the intended use and if they are validated. The client shall be informed as to the method chosen.

d) The laboratory shall inform the client when the method proposed by the client is considered to be inappropriate or out of date.

5.5.4.2.2 Demonstration of Capability

The laboratory shall confirm that it can properly operate all methods before introducing the environmental tests ~~or calibrations.~~ If the method changes, the confirmation shall be repeated.

a) Prior to acceptance and institution of any method, satisfactory demonstration of method capability is required. (See Appendix C and 5.5.2.6.b) In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean quality system matrix sample of a quality system matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., drinking water, solids, biological tissue and air. In addition, for analytes which do not lend themselves to spiking, the demonstration of capability may be performed using quality control samples.

b) Thereafter, continuing demonstration of method performance, as per the quality control requirements in Appendix D (such as laboratory control samples) is required.

c) In cases where a laboratory analyzes samples using a method that has been in use by the laboratory before July 1999, and there have been no significant changes in instrument type, personnel or method, the continuing demonstration of method performance and the analyst's documentation of continued proficiency shall be acceptable. The laboratory shall have records on file to demonstrate that a demonstration of capability is not required.

d) In all cases, the appropriate forms such as the Certification Statement (Appendix C) must be completed and retained by the laboratory to be made available upon request. All associated supporting data necessary to reproduce the analytical results summarized in the Certification Statement must be retained by the laboratory. (See Appendix C for Certification Statement.)

e) A demonstration of capability must be completed each time there is a change in instrument type, personnel, or method.

f) In laboratories with a specialized "work cell(s)" (a group consisting of analysts with specifically defined tasks that together perform the test method), the group as a unit must meet the above criteria and this demonstration of capability must be fully documented.

g) When a work cell(s) is employed, and the members of the cell change, the new employee(s) must work with experienced analyst(s) in that area of the work cell where they are employed. This new work cell must demonstrate acceptable performance through acceptable continuing performance checks (appropriate sections of Appendix D, such as laboratory control samples). Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability

must be repeated. In addition, if the entire work cell is changed/replaced, the work cell must perform the demonstration of capability (Appendix C).

h) When a work cell(s) is employed the performance of the group must be linked to the training record of the individual members of the work cell (see section 5.5.2.6).

5.5.4.3 Laboratory-Developed Methods

The introduction of environmental test ~~and calibration~~ methods developed by the laboratory for its own use shall be a planned activity and shall be assigned to qualified personnel equipped with adequate resources.

Plans shall be updated as development proceeds and effective communication amongst all personnel involved shall be ensured.

5.5.4.4 Non-Standard Methods

When it is necessary to use methods not covered by standard methods, these shall be subject to agreement with the client and shall include a clear specification of the client's requirements and the purpose of the environmental test ~~and/or calibration~~. The method developed shall have been validated appropriately before use.

5.5.4.5 Validation of Methods

5.5.4.5.1 Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

5.5.4.5.2 The laboratory shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their published ~~intended~~ scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application. The laboratory shall record the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use. The minimum requirements shall be the initial test method evaluation requirements given in Appendix C.3 of this chapter.

5.5.4.5.3 The range and accuracy of the values obtainable from validated methods (e. g. the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), as assessed for the intended use, shall be relevant to the clients' needs.

5.5.4.6 Estimation of Uncertainty of Measurement

~~**5.5.4.6.1** A calibration laboratory, or an environmental testing laboratory performing its own calibrations and issuing a calibration certificate, shall have and shall apply a procedure to estimate the uncertainty of measurement for all calibrations and types of calibrations.~~

~~**5.5.4.6.2**~~**5.5.4.6.1** Environmental testing laboratories shall have and shall apply procedures for estimating uncertainty of measurement. In certain cases the nature of the test method may preclude rigorous, metrologically and statistically valid, calculation of uncertainty of measurement. In these cases the laboratory shall at least attempt to identify all the components of uncertainty and make a reasonable estimation, and shall ensure that the form of reporting of the result does not give a wrong

impression of the uncertainty. Reasonable estimation shall be based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data.

In those cases where a well-recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied this clause by following the test method and reporting instructions (see 5.5.10).

~~**5.5.4.6.3**~~**5.5.4.6.2** When estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation shall be taken into account using appropriate methods of analysis.

5.5.4.7 Control of Data

5.5.4.7.1 Calculations and data transfers shall be subject to appropriate checks in a systematic manner.

a) The laboratory shall establish SOPs to ensure that the reported data are free from transcription and calculation errors.

b) The laboratory shall establish SOPs to ensure that all quality control measures are reviewed, and evaluated before data are reported.

c) The laboratory shall establish SOPs addressing manual calculations including manual integrations.

5.5.4.7.2 When computers, automated equipment, or microprocessors are used for the acquisition, processing, recording, reporting, storage or retrieval of environmental test ~~or calibration~~ data, the laboratory shall ensure that:

a) computer software developed by the user is documented in sufficient detail and is suitably validated as being adequate for use;

b) procedures are established and implemented for protecting the data; such procedures shall include, but not be limited to, integrity and confidentiality of data entry or collection, data storage, data transmission and data processing;

c) computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of environmental test ~~and calibration~~ data.

d) it establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.

Commercial off-the-shelf software (e. g. word processing, database and statistical programs) in general use within their designed application range is considered to be sufficiently validated. However, laboratory software configuration or modifications must be validated as in 5.5.4.7.2a.

5.5.5 Equipment

5.5.5.1 The laboratory shall be furnished with all items of sampling, measurement and test equipment required for the correct performance of the environmental tests ~~and/or calibrations~~ (including sampling, preparation of samples, processing and analysis of environmental ~~test and/or calibration~~ data). In those cases where the laboratory needs to use equipment outside its permanent control, it shall ensure that the requirements of this Standard are met.

5.5.5.2 Equipment and its software used for testing~~, calibration~~ and sampling shall be capable of achieving the accuracy required and shall comply with specifications relevant to the environmental tests ~~and/or calibrations~~ concerned. ~~Calibration programs shall be established for key quantities or values of the instruments where these properties have a significant effect on the results.~~ Before being placed into service, equipment (including that used for sampling) shall be calibrated or checked to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications~~. It shall be checked and/or calibrated before use (see 5.5.6).~~

Calibration requirements are divided into two parts: (1) requirements for analytical support equipment, and 2) requirements for instrument calibration. In addition, the requirements for instrument calibration are divided into initial instrument calibration and continuing instrument calibration verification.

5.5.5.2.1 Support Equipment

These standards apply to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume.

a) All support equipment shall be maintained in proper working order. The records of all repair and maintenance activities including service calls, shall be kept.

b) All support equipment shall be calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration or verification shall be within the specifications required of the application for which this equipment is used or:

   1) the equipment shall be removed from service until repaired; or

   2) the laboratory shall maintain records of established correction factors to correct all measurements.

c) Raw data records shall be retained to document equipment performance.

d) Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths shall be checked in the expected use range, with NIST traceable references where commercially available. The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

e) Mechanical volumetric dispensing devices including burettes (except Class A glassware) shall be checked for accuracy on at least a quarterly use basis. Glass microliter syringes are to be considered in the same manner as Class A glassware, but must come with a certificate attesting to established accuracy or the accuracy must be initially demonstrated and documented by the laboratory.

f) For chemical tests the temperature, cycle time, and pressure of each run of autoclaves must be documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges.

g) For biological tests that employ autoclave sterilization see section D.3.8.

5.5.5.2.2 Instrument Calibration

This standard specifies the essential elements that shall define the procedures and documentation for initial instrument calibration and continuing instrument calibration verification to ensure that the data must be of known quality and be appropriate for a given regulation or decision. This standard does not specify detailed procedural steps ("how to") for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not apparent which standard is more stringent, then the requirements of the regulation or mandated test method are to be followed.

~~**Note: In the following sections, initial instrument calibration is directly used for quantitation and continuing instrument calibration verification is used to confirm the continued validity of the initial calibration unless otherwise required by regulation, method, or program.**~~

5.5.5.2.2.1 Initial Instrument Calibration

The following items are essential elements of initial instrument calibration:

a) The details of the initial instrument calibration procedures including calculations, integrations, acceptance criteria and associated statistics must be included or referenced in the test method SOP. When initial instrument calibration procedures are referenced in the test method, then the referenced material must be retained by the laboratory and be available for review.

b) Sufficient raw data records must be retained to permit reconstruction of the initial instrument calibration, e.g., calibration date, test method, instrument, analysis date, each analyte name, analyst's initials or signature; concentration and response, calibration curve or response factor; or unique equation or coefficient used to reduce instrument responses to concentration.

c) Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method, or program.

d) All initial instrument calibrations must be verified with a standard obtained from a second manufacturer or lot if the lot can be demonstrated from the manufacturer as prepared independently from other lots. Traceability shall be to a national standard, when commercially available.

e) Criteria for the acceptance of an initial instrument calibration must be established, e.g., correlation coefficient or relative percent difference. The criteria used must be appropriate to the calibration technique employed.

f) The lowest calibration standard concentration for purposes of establishing the working calibration range shall be the lower limit of quantitation. Any data reported below the lower limit of quantitation should be considered to have an increased quantitative uncertainty and shall be reported using defined qualifiers or flags or explained in the case narrative.

g) The highest calibration standard shall be the highest concentration for which quantitative data are to be reported (see Appendix C.) Any data reported above this highest standard should be considered to have an increased quantitative uncertainty and shall be reported using defined qualifiers or flags or explained in the case narrative.

~~f)~~h) Measured concentrations outside the working range shall be reported as having less certainty and shall be reported using defined qualifiers or flags or explained in the case narrative. ~~Results of samples outside of the concentration range established by the initial calibration must be reported with defined qualifiers or flags or explained in the case narrative.~~ The lowest calibration standard must be above the limit of detection ~~detection limit~~. Noted exception: The following shall occur for instrument technology (such as ICP or ICP/MS) with validated techniques from manufacturers or methods employing standardization with a zero point and a single point calibration standard:

1) Prior to the analysis of samples the zero point and single point calibration must be analyzed and the linear range of the instrument must be established by analyzing a series of standards, one of which must be at the lowest quantitation level. Sample results within the established linear range will not require data qualifier flags.

2) Zero point and single point calibration standard must be analyzed with each analytical batch.

3) A standard corresponding to the lowest quantitation level must be analyzed with each analytical batch and must meet established acceptance criteria.

4) The linearity is verified at a frequency established by the method and/or the manufacturer.

~~5) If a sample within an analytical batch produces results above its associated single point standard then one of the following should occur:~~

~~i) analyze reference material at or above the sample value that meets established acceptance criteria for validating the linearity;~~
~~ii) dilute the sample such that the result falls below the single point calibration concentration;~~
~~iii) report the data with an appropriate data qualifier and/or explain in the case narrative.~~

~~g)~~i) If the initial instrument calibration results are outside established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed. If reanalysis of the samples is not possible, data associated with an unacceptable initial instrument calibration shall be reported with appropriate data qualifiers.

~~h) Calibration standards must include concentrations at or below the regulatory limit/decision level, if these limits/levels are known by the laboratory, unless these concentrations are below the laboratory's demonstrated detection limits (See D.1.4 Detection Limits)~~

~~i)~~j) If a reference or mandated method does not specify the number of calibration standards, the minimum number is two, (one of which must be at the lowest limit of quantitation ~~quantitation limit~~) not including blanks or a zero standard with the noted exception of instrument technology for which it has been established by methodologies and procedures that a zero and a single point standard are appropriate for calibrations (see 5.5.5.2.2.1 h)~~5.9.4.2.1.f~~). The laboratory must have a standard operating procedure for determining the number of points for establishing the initial instrument calibration.

5.5.5.3 Equipment shall be operated by authorized personnel. Up-to-date instructions on the use and maintenance of equipment (including any relevant manuals provided by the manufacturer of the equipment) shall be readily available for use by the appropriate laboratory personnel.

All equipment shall be properly maintained, inspected and cleaned. Maintenance procedures shall be documented.

5.5.5.4 Each item of equipment and its software used for environmental testing ~~and calibration~~ and significant to the result shall, when practicable, be uniquely identified.

5.5.5.5 ~~Records shall be maintained~~ The laboratory shall maintain records of each major item of equipment and its software significant to the environmental tests ~~and/or calibrations~~ performed. The records shall include at least the following:

a) the identity of the item of equipment and its software;

b) the manufacturer's name, type identification, and serial number or other unique identification;

c) checks that equipment complies with the specification (see 5.5.5.2);

d) the current location~~, where appropriate~~;

e) the manufacturer's instructions, if available, or reference to their location;

f) dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration;

g) the maintenance plan, where appropriate, and maintenance carried out to date; documentation on all routine and non-routine maintenance activities and reference material verifications.

h) any damage, malfunction, modification or repair to the equipment.

i) date received and date placed in service (if available);

j) if available, condition when received (e.g. new, used, reconditioned);

5.5.5.6 The laboratory shall have procedures for safe handling, transport, storage, use and planned maintenance of measuring equipment to ensure proper functioning and in order to prevent contamination or deterioration.

5.5.5.7 Equipment that has been subjected to overloading or mishandling, gives suspect results, or has been shown to be defective or outside specified limits, shall be taken out of service. It shall be isolated to prevent its use or clearly labeled or marked as being out of service, until it has been

repaired and shown by calibration or test to perform correctly. The laboratory shall examine the effect of the defect or departure from specified limits on previous environmental tests ~~and/or calibrations~~ and shall institute the "Control of nonconforming work" procedure (see 5.4.9).

5.5.5.8 Whenever practicable, all equipment under the control of the laboratory and requiring calibration shall be labeled, coded or otherwise identified to indicate the status of calibration, including the date when last calibrated and the date or expiration criteria when recalibration is due.

5.5.5.9 When, for whatever reason, equipment goes outside the direct control of the laboratory, the laboratory shall ensure that the function and calibration status of the equipment are checked and shown to be satisfactory before the equipment is returned to service.

5.5.5.10 ~~When intermediate checks are needed to maintain confidence in the calibration status of the equipment, these checks shall be carried out according to a defined procedure.~~

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration shall be verified prior to sample analyses by continuing instrument calibration verification with each analytical batch. The following items are essential elements of continuing instrument calibration verification:

a) The details of the continuing instrument calibration procedure, calculations and associated statistics must be included or referenced in the test method SOP.

b) ~~A continuing instrument calibration verification must be repeated at the beginning and end of each analytical batch. The concentrations of the calibration verification shall be varied within the established calibration range. If an internal standard is used, only one continuing instrument calibration verification must be analyzed per analytical batch.~~Calibration shall be verified for each compound, element, or other discrete chemical species, except for mixtures such as Arolor-1254, Total Petroleum Hydrocarbons, or Toxaphene where a representative chemical related substance or mixture can be used.

c) Instrument calibration verification must be performed:

1) at the beginning and end of each analytical batch (except, if an internal standard is used, only one verification needs to be performed at the beginning of the analytical batch);

2) whenever it is expected that the analytical system may be out of calibration or might not meet the verification acceptance criteria;

3) if the time period for calibration or the most previous calibration verification has expired; or

4) for analytical systems that contain a calibration verification requirement.

~~c)~~d) Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations. Continuing calibration verification records must explicitly connect the continuing verification data to the initial instrument calibration.

~~d)~~e) Criteria for the acceptance of a continuing instrument calibration verification must be established, e.g., relative percent difference.

e) If the continuing instrument calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive ~~successful~~ calibration verifications, or a new initial instrument calibration must be performed. ~~If the laboratory has not demonstrated acceptable performance, sample analyses shall not occur until a new initial calibration curve is established and verified. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions~~ If the laboratory has not verified calibration, sample analyses may not occur until the analytical system is calibrated or calibration verified. If samples are analyzed using a system on which the calibration has not yet been verified the results shall be flagged. Data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

1) when the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

2) when the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

5.5.5.11 Where calibrations give rise to a set of correction factors, the laboratory shall have procedures to ensure that copies (e. g. in computer software) are correctly updated.

5.5.5.12 Test ~~and calibration~~ equipment, including both hardware and software, shall be safeguarded from adjustments which would invalidate the test ~~and/or calibration~~ results.

5.5.6 Measurement Traceability

5.5.6.1 General

All equipment used for environmental tests ~~and/or calibrations~~, including equipment for subsidiary measurements (e. g. for environmental conditions) having a significant effect on the accuracy or validity of the result of the environmental test~~, calibration~~ or sampling shall be calibrated before being put into service and on a continuing basis. The laboratory shall have an established program and procedure for the calibration of its equipment. This includes balances, thermometers, and control standards. Such a program shall include a system for selecting, using, calibrating, checking, controlling and maintaining measurement standards, reference materials used as measurement standards, and measuring and test equipment used to perform environmental tests ~~and calibrations~~.

5.5.6.2 ~~Specific Requirements~~ Testing Laboratories

5.5.6.2.1 ~~Calibration Laboratories~~

~~For the purpose of this Standard, a calibration laboratory is a laboratory that issues a calibration certificate.~~

~~**5.5.6.2.1.1** For calibration laboratories, the program for calibration of equipment shall be designed and operated so as to ensure that calibrations and measurements made by the laboratory are traceable to the International System of Units (SI).~~

~~A calibration laboratory establishes traceability of its own measurement standards and measuring instruments to the SI by means of an unbroken chain of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement. The link to SI units may be achieved by reference to national measurement standards. National measurement standards may be primary standards, which are primary realizations of the SI units or agreed representations of SI units based on fundamental physical constants, or they may be secondary standards which are standards calibrated by another national metrology institute. When using external calibration services, traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability. The calibration certificates issued by these laboratories shall contain the measurement results, including the measurement uncertainty and/or a statement of compliance with an identified metrological specification (see also 5.5.10.4.2).~~

~~**5.5.6.2.1.2** There are certain calibrations that currently cannot be strictly made in SI units. In these cases calibration shall provide confidence in measurements by establishing traceability to appropriate measurement standards such as:~~

~~a) the use of certified reference materials provided by a competent supplier to give a reliable physical or chemical characterization of a material;~~

~~b) the use of specified methods and/or consensus standards that are clearly described and agreed by all parties concerned.~~

~~Participation in a suitable program of interlaboratory comparisons is required where possible.~~

~~5.5.6.2.2 Testing Laboratories~~

~~5.5.6.2.2.1~~ For testing laboratories, ~~the requirements given in 5.5.6.2.1 apply for measuring and test equipment with measuring functions used, unless it has been established that the associated contribution from the calibration contributes little to the total uncertainty of the test result. When this situation arises,~~ the laboratory shall ensure that the equipment used can provide the uncertainty of measurement needed.

a) The overall program of calibration and/or verification and validation of equipment shall be designed and operated so as to ensure that measurements made by the laboratory are traceable to national standards of measurement.

5.5.6.2.2.~~2~~ Where traceability of measurements to SI units is not possible ~~and/~~or not relevant, the same requirements for traceability to, for example, certified reference materials, agreed methods and/or consensus standards, are required~~ as for calibration laboratories (see 5.5.6.2.1.2).~~ The laboratory shall provide satisfactory evidence of correlation of results, for example by participation in a suitable program of interlaboratory comparisons, proficiency testing, or independent analysis.

~~a) The overall program of calibration and/or verification and validation of equipment shall be designed and operated so as to ensure that measurements made by the laboratory are traceable to national standards of measurement.~~

~~b) Calibration certificates shall indicate the traceability to national standards of measurement and shall provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The laboratory shall maintain records of all such certifications.~~

~~c) Where traceability to national standards of measurement is not applicable, the laboratory shall provide satisfactory evidence of correlation of results, for example by participation in a suitable program of interlaboratory comparisons, proficiency testing, or independent analysis.~~

5.5.6.3 Reference Standards and Reference Materials

5.5.6.3.1 Reference Standards

The laboratory shall have a program and procedure for the calibration of its reference standards. Reference standards shall be calibrated by a body that can provide traceability as described in 5.5.6.2.1. Such reference standards of measurement held by the laboratory (such as class S or equivalent weights or traceable thermometers) shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated. Reference standards shall be calibrated before and after any adjustment. Where commercially available ~~possible~~, this traceability shall be to a national standard of measurement.

5.5.6.3.2 Reference Materials

Reference materials shall, where commercially available ~~possible~~, be traceable to SI units of measurement, or to certified reference materials. Where possible, traceability shall be to national or international standards of measurement, or to national or international standard reference materials. Internal reference materials shall be checked as far as is technically and economically practicable.

5.5.6.3.3 Intermediate Checks

Checks needed to maintain confidence in the ~~calibration~~ status of reference, primary, transfer or working standards and reference materials shall be carried out according to defined procedures and schedules.

5.5.6.3.4 Transport and Storage

The laboratory shall have procedures for safe handling, transport, storage and use of reference standards and reference materials in order to prevent contamination or deterioration and in order to protect their integrity.

5.5.6.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Documented procedures shall exist for the purchase, reception and storage of consumable materials used for the technical operations of the laboratory.

a) The laboratory shall retain records for all standards, reagents, reference materials and media including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if

supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material shall not be used unless its reliability is verified by the laboratory.

b) Original containers (such as provided by the manufacturer or vendor) shall be labeled with an expiration date.

c) Records shall be maintained on ~~reagent,~~ standard~~,~~ and reference material preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials.

d) All containers of prepared ~~reagents,~~ standards~~,~~ and reference materials must bear a unique identifier and expiration date and be linked to the documentation requirements in 5.5.6.4.c above.

e) Procedures shall be in place to ensure prepared reagents meet the requirements of the test method. The source of reagents shall comply with 5.5.9.2 a) 6) and D.1.4 b).

f) All containers of prepared reagents must bear a preparation date. An expiration date shall be defined on the container or documented elsewhere as indicated in the laboratory's quality manual or SOP.

5.5.7 Sampling

5.5.7.1 The laboratory shall have a sampling plan and procedures for sampling when it carries out sampling of substances, materials or products for subsequent environmental testing ~~or calibration~~. The sampling plan as well as the sampling procedure shall be available at the location where sampling is undertaken. Sampling plans shall, whenever reasonable, be based on appropriate statistical methods. The sampling process shall address the factors to be controlled to ensure the validity of the environmental test ~~and calibration~~ results.

Where sampling (as in obtaining sample aliquots from a submitted sample) is carried out as part of the test method, the laboratory shall use documented procedures and appropriate techniques to obtain representative subsamples.

5.5.7.2 Where the client requires deviations, additions or exclusions from the documented sampling procedure, these shall be recorded in detail with the appropriate sampling data and shall be included in all documents containing environmental test and/or calibration results, and shall be communicated to the appropriate personnel.

5.5.7.3 The laboratory shall have procedures for recording relevant data and operations relating to sampling that forms part of the environmental testing ~~or calibration~~ that is undertaken. These records shall include the sampling procedure used, the identification of the sampler, environmental conditions (if relevant) and diagrams or other equivalent means to identify the sampling location as necessary and, if appropriate, the statistics the sampling procedures are based upon.

5.5.8 Handling of Samples

While the laboratory may not have control of field sampling activities, the following are essential to ensure the validity of the laboratory's data.

5.5.8.1 The laboratory shall have procedures for the transportation, receipt, handling, protection, storage, retention and/or disposal of samples, including all provisions necessary to protect the integrity of the sample, and to protect the interests of the laboratory and the client.

5.5.8.2 The laboratory shall have a system for identifying samples. The identification shall be retained throughout the life of the sample in the laboratory. The system shall be designed and operated so as to ensure that samples cannot be confused physically or when referred to in records or other documents. The system shall, if appropriate, accommodate a sub-division of groups of samples and the transfer of samples within and from the laboratory.

a) The laboratory shall have a documented system for uniquely identifying the samples to be tested, to ensure that there can be no confusion regarding the identity of such samples at any time. This system shall include identification for all samples, subsamples and subsequent extracts and/or digestates. The laboratory shall assign a unique identification (ID) code to each sample container received in the laboratory. The use of container shape, size or other physical characteristic, such as amber glass, or purple top, is not an acceptable means of identifying the sample.

b) This laboratory code shall maintain an unequivocal link with the unique field ID code assigned each container.

c) The laboratory ID code shall be placed on the sample container as a durable label.

d) The laboratory ID code shall be entered into the laboratory records (see 5.5.8.3.1.d) and shall be the link that associates the sample with related laboratory activities such as sample preparation ~~or calibration~~.

e) In cases where the sample collector and analyst are the same individual, or the laboratory preassigns numbers to sample containers, the laboratory ID code may be the same as the field ID code.

5.5.8.3 Upon receipt of the samples, the condition, including any abnormalities or departures from normal or specified conditions as described in the environmental test ~~or calibration~~ method, shall be recorded. When there is doubt as to the suitability of a sample for environmental test ~~or calibration~~, or when a sample does not conform to the description provided, or the environmental test ~~or calibration~~ required is not specified in sufficient detail, the laboratory shall consult the client for further instructions before proceeding and shall record the discussion.

5.5.8.3.1 Sample Receipt Protocols

a) All items specified in 5.5.8.3.2 below shall be checked.

1) All samples which require thermal preservation shall be considered acceptable if the arrival temperature is either within 2°C of the required temperature or the method specified range. For samples with a specified temperature of 4°C, samples with a temperature ranging from just above the freezing temperature of water to 6°C shall be acceptable. Samples that are hand delivered to the laboratory on the same day that they are collected ~~immediately after collection~~ may not meet these ~~this~~ criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

2) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or chlorine, prior to or during sample preparation or analysis.

3) Microbiological samples from chlorinated water systems do not require an additional chlorine residual check in the laboratory if the following conditions are met:

   i. sufficient sodium thiosulfate is added to each container to neutralize at minimum 5 mg/l of chlorine for drinking water and 15mg/l of chlorine for wastewater samples;

   ii. one container from each batch of laboratory prepared containers or lot of purchased ready-to-use containers is checked to ensure efficacy of the sodium thiosulfate to 5 mg/l chlorine or 15mg/l chlorine as appropriate and the check is documented;

   iii. chlorine residual is checked in the field and actual concentration is documented with sample submission.

b) The results of all checks shall be recorded.

c) If the sample does not meet the sample receipt acceptance criteria listed in this standard, the laboratory shall either:

1) retain correspondence and/or records of conversations concerning the final disposition of rejected samples; or

2) fully document any decision to proceed with the analysis of samples not meeting acceptance criteria.

   i. The condition of these samples shall, at a minimum, be noted on the chain of custody or transmittal form and laboratory receipt documents.

   ii. The analysis data shall be appropriately "qualified" on the final report.

d) The laboratory shall utilize a permanent chronological record such as a log book or electronic database to document receipt of all sample containers.

1) This sample receipt log shall record the following:

   i. client/project name,

   ii. date and time of laboratory receipt,

   iii. unique laboratory ID code (see 5.5.8.2), and,

   iv. signature or initials of the person making the entries.

2) During the log-in process, the following information must be unequivocally linked to the log record or included as a part of the log. If such information is recorded/documented elsewhere, the records shall be part of the laboratory's permanent records, easily retrievable upon request and readily available to individuals who will process the sample. Note: the placement of the laboratory ID number on the sample container is not considered a permanent record.

   i. The field ID code which identifies each container must be linked to the laboratory ID code in the sample receipt log.

ii. The date and time of sample collection must be linked to the sample container and to the date and time of receipt in the laboratory.

iii. The requested analyses (including applicable approved test method numbers) must be linked to the laboratory ID code.

iv. Any comments resulting from inspection for sample rejection shall be linked to the laboratory ID code.

e) All documentation, such as memos or transmittal forms, that is transmitted to the laboratory by the sample transmitter shall be retained.

f) A complete chain of custody record form (Sections 5.4.12.2.5 and Appendix E), if utilized, shall be maintained.

5.5.8.3.2 Sample Acceptance Policy

The laboratory must have a written sample acceptance policy that clearly outlines the circumstances under which samples shall be accepted or rejected. Data from any samples which do not meet the following criteria must be flagged in an unambiguous manner clearly defining the nature and substance of the variation. This sample acceptance policy shall be made available to sample collection personnel and shall include, but is not limited to, the following areas of concern:

a) proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;

b) proper sample labeling to include unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;

c) use of appropriate sample containers;

d) adherence to specified holding times;

e) adequate sample volume. Sufficient sample volume must be available to perform the necessary tests; and

f) procedures to be used when samples show signs of damage, contamination or inadequate preservation.

5.5.8.4 The laboratory shall have procedures and appropriate facilities for avoiding deterioration, contamination, loss or damage to the sample during storage, handling, preparation and testing. Handling instructions provided with the sample shall be followed. When samples have to be stored or conditioned under specified environmental conditions, these conditions shall be maintained, monitored and recorded. Where a sample or a portion of a sample is to be held secure, the laboratory shall have arrangements for storage and security that protect the condition and integrity of the secured samples or portions concerned.

a) Samples shall be stored according to the conditions specified by preservation protocols:

1) Samples which require thermal preservation shall be stored under refrigeration which is +/-2 of the specified preservation temperature unless method specific criteria exist. For

samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C shall be acceptable.

2) Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in such a manner to prevent cross contamination.

b) Sample fractions, extracts, leachates and other sample preparation products shall be stored according to 5.5.8.4.a above or according to specifications in the test method.

d) The laboratory shall have SOPs for the disposal of samples, digestates, leachates and extracts or other sample preparation products.

5.5.9 Assuring the Quality of Environmental Test and Calibration Results

5.5.9.1 General

The laboratory shall have quality control procedures for monitoring the validity of environmental tests ~~and calibrations~~ undertaken. The resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results. This monitoring shall be planned and reviewed and may include, but not be limited to, the following:

a) regular use of certified reference materials and/or internal quality control using secondary reference materials;

b) participation in interlaboratory comparison or proficiency-testing program (see Chapter 2)

c) replicate tests ~~or calibrations~~ using the same or different methods;

d) retesting ~~or recalibration~~ of retained samples;

e) correlation of results for different characteristics of a sample (for example, total phosphate should be greater than or equal to orthophosphate).

5.5.9.2 Essential Quality Control Procedures

These general quality control principles shall apply, where applicable, to all testing laboratories. The manner in which they are implemented is dependent on the types of tests performed by the laboratory (i.e., chemical, whole effluent toxicity, microbiological, radiological, air) and are further described in Appendix D. The standards for any given test type shall assure that the applicable principles are addressed:

a) All laboratories shall have detailed written protocols in place to monitor the following quality controls:

1) positive and negative controls to monitor tests such as blanks, spikes, reference toxicants;

2) tests to define the variability and/or repeatability of the laboratory results such as replicates;

3) measures to assure the accuracy of the test method including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

4) measures to evaluate test method capability, such as limit of detection ~~detection limit~~s and limit of quantitation ~~quantitation limits~~ or range of applicability such as linearity;

5) selection of appropriate formulae to reduce raw data to final results such as regression analysis, comparison to internal/external standard calculations, and statistical analyses;

6) selection and use of reagents and standards of appropriate quality;

7) measures to assure the selectivity of the test for its intended purpose; and

8) measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the test method such as temperature, humidity, light, or specific instrument conditions.

b) All quality control measures shall be assessed and evaluated on an on-going basis, and quality control acceptance criteria shall be used to determine the usability of the data. (See Appendix D.)

c) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist. (See 5.5.8.3.2, Sample Acceptance Policy.)

d) The quality control protocols specified by the laboratory's method manual (5.5.4.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D or mandated methods or regulations (whichever are more stringent) are incorporated into their method manuals. When it is not apparent which is more stringent the QC in the mandated method or regulations is to be followed.

The essential quality control measures for testing are found in Appendix D of this Chapter.

5.5.10 Reporting the Results

5.5.10.1 General

The results of each test, ~~calibration,~~ or series of environmental tests ~~or calibrations~~ carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively, and in accordance with any specific instructions in the environmental test or calibration methods.

The results shall be reported, ~~usually~~ in a test report ~~or a calibration certificate,~~ and shall include all the information requested by the client and necessary for the interpretation of the environmental test or calibration results and all information required by the method used. This information is normally that required by 5.5.10.2, and 5.5.10.3 ~~or 5.5.10.4~~.

In the case of environmental tests ~~or calibrations~~ performed for internal clients, or in the case of a written agreement with the client, the results may be reported in a simplified way. Any information listed in 5.5.10.2 to 5.5.10.4 which is not reported to the client shall be readily available in the laboratory which carried out the environmental tests ~~and/or calibrations~~.

Some regulatory reporting requirements or formats such as monthly operating reports may not require all items listed below, however, the laboratory shall provide all the required information to their client for use in preparing such regulatory reports.

Laboratories that are operated by a facility and whose sole function is to provide data to the facility management for compliance purposes (in-house or captive laboratories) shall have all applicable information specified in a) through m) below readily available for review by the accrediting authority. However, formal reports detailing the information are not required if:

a) the in-house laboratory is itself responsible for preparing the regulatory reports; or

b) the laboratory provides information to another individual within the organization for preparation of regulatory reports. The facility management must ensure that the appropriate report items are in the report to the regulatory authority if such information is required.

5.5.10.2 Test Reports ~~and Calibration Certificates~~

Each test report ~~or calibration certificate~~ shall include at least the following information, unless the laboratory has valid reasons for not doing so, as indicated by 5.5.10.1.a and b:

a) a title (e.g. "Test Report," ~~"Calibration Certificate,"~~ "Certificate of Results," or "Laboratory Results");

b) the name and address of the laboratory, the location where the environmental tests ~~and/or calibrations~~ were carried out, if different from the address of the laboratory, and phone number with name of contact person for questions;

c) unique identification of the test report ~~or calibration certificate~~ (such as the serial number), and on each page an identification in order to ensure that the page is recognized as a part of the test report ~~or calibration certificate~~, and a clear identification of the end of the test report ~~or calibration certificate~~;

    1) This requirement may be presented in several ways:

        i. The total number of pages may be listed on the first page of the report as long as the subsequent pages are identified by the unique report identification and consecutive numbers, or

        ii. Each page is identified with the unique report identification. The pages are identified as a number of the total report pages (example: 3 of 10, or 1 of 20).

    2) Other methods of identifying the pages in the report may be acceptable as long as it is clear to the reader that discrete pages are associated with a specific report, and that the report contains a specified number of pages.

d) the name and address of the client and project name if applicable;

e) identification of the method used;

f) a description of, the condition of, and unambiguous identification of the sample(s), including the client identification code;

g) the date of receipt of the sample(s) where this is critical to the validity and application of the results, date and time of sample collection, the date(s) of performance of the environmental test ~~or calibration~~, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 72 hours;

h) reference to the sampling plan and procedures used by the laboratory or other bodies where these are relevant to the validity or application of the results;

i) the environmental test ~~or calibration~~ results with, where appropriate, the units of measurement, and any failures identified; identify whether data are calculated on a dry weight or wet weight basis; identify the reporting units such as ì g/l or mg/kg; and for Whole Effluent Toxicity, identify the statistical package used to provide data;

j) the name(s), function(s) and signature(s) or equivalent electronic identification of person(s) authorizing the test report ~~or calibration certificate~~, and date of issue;

k) ~~where relevant,~~ a statement to the effect that the results relate only to the samples;

l) at the laboratory's discretion, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory;

m) Laboratories accredited to be in compliance with these standards shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

5.5.10.3 Supplemental Information for Test Reports

5.5.10.3.1 In addition to the requirements listed in 5.5.10.2, test reports shall, where necessary for the interpretation of the test results, include the following:

a) deviations from (such as failed quality control), additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions and any non-standard conditions that may have affected the quality of results, including the use and definitions of data qualifiers;

b) where quality system requirements are not met~~relevant~~, a statement of compliance/non-compliance with requirements and/or specifications, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature;

c) where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed ~~in test reports when it is relevant to the validity or application of the test results,~~ when a client's instruction so requires~~, or when the uncertainty affects compliance to a specification limit~~;

d) where appropriate and needed, opinions and interpretations (see 5.5.10.4 ~~5.5.10.5~~);

e) additional information which may be required by specific methods, clients or groups of clients;

f) clear identification of numerical results with values outside of limits of quantitaion ~~quantitation limits~~.

5.5.10.3.2 In addition to the requirements listed in 5.5.10.2 and 5.5.10.3.1, test reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

a) the date of sampling;

b) unambiguous identification of the substance, material or product sampled (including the name of the manufacturer, the model or type of designation and serial numbers as appropriate);

c) the location of sampling, including any diagrams, sketches or photographs;

d) a reference to the sampling plan and procedures used;

e) details of any environmental conditions during sampling that may affect the interpretation of the test results;

f) any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

5.5.10.4 ~~Calibration Certificates~~

~~**5.5.10.4.1** In addition to the requirements listed in 5.5.10.2, calibration certificates shall include the following, where necessary for the interpretation of calibration results:~~

~~a) the conditions (e.g. environmental) under which the calibrations were made that have an influence on the measurement results;~~

~~b) the uncertainty of measurement and/or a statement of compliance with an identified metrological specification or clauses thereof;~~

~~c) evidence that the measurements are traceable.~~

~~**5.5.10.4.2** The calibration certificate shall relate only to quantities and the results of functional tests. If a statement of compliance with a specification is made, this shall identify which clauses of the specification are met or not met.~~

~~When a statement of compliance with a specification is made omitting the measurement results and associated uncertainties, the laboratory shall record those results and maintain them for possible future reference.~~

~~When statements of compliance are made, the uncertainty of measurement shall be taken into account.~~

~~**5.5.10.4.3** When an instrument for calibration has been adjusted or repaired, the calibration results before and after adjustment or repair, if available, shall be reported.~~

~~**5.5.10.4.4** A calibration certificate (or calibration label) shall not contain any recommendation on the calibration interval except where this has been agreed with the client. This requirement may be superseded by legal regulations.~~

~~5.5.10.5~~ Opinions and Interpretations

When opinions and interpretations are included, the laboratory shall document the basis upon which the opinions and interpretations have been made. Opinions and interpretations shall be clearly marked as such in a test report.

5.5.10.~~6~~5 Environmental Testing ~~and Calibration Results~~ Obtained from Subcontractors

When the test report contains results of tests performed by subcontractors, these results shall be clearly identified by subcontractor name or applicable accreditation number. The subcontractor shall report the results in writing or electronically. The laboratory shall make a copy of the subcontractor's report available to the client when requested by the client.

~~When a calibration has been subcontracted, the laboratory performing the work shall issue the calibration certificate to the contracting laboratory.~~

5.5.10.~~7~~6 Electronic Transmission of Results

In the case of transmission of environmental test ~~or calibration~~ results by telephone, telex, facsimile or other electronic or electromagnetic means, the requirements of this Standard shall be met and ensure that all reasonable steps are taken to preserve confidentiality (see also 5.5.4.7).

5.5.10.~~8~~7 Format of Reports and Certificates

The format shall be designed to accommodate each type of environmental test ~~or calibration~~ carried out and to minimize the possibility of misunderstanding or misuse.

5.5.10.~~9~~8 Amendments to Test Reports and Calibration Certificates

Material amendments to a test report ~~or calibration~~ certificate after issue shall be made only in the form of a further document, or data transfer, which includes the statement:

"Supplement to Test Report ~~[or Calibration Certificate]~~, serial number ... [or as otherwise identified]", or an equivalent form of wording.

Such amendments shall meet all the requirements of this Standard.

When it is necessary to issue a complete new test report ~~or calibration certificate~~, this shall be uniquely identified and shall contain a reference to the original that it replaces.

QUALITY SYSTEMS
APPENDIX A

REFERENCES

Appendix A - REFERENCES

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APPENDIX B--(Reserved)

QUALITY SYSTEMS
APPENDIX C

DEMONSTRATION OF CAPABILITY

Appendix C - DEMONSTRATION OF CAPABILITY

C.1 PROCEDURE FOR DEMONSTRATION OF CAPABILITY

A demonstration of capability (DOC) must be made prior to using any test method, and at any time there is a change in instrument type, personnel or test method (see 5.5.4.2.2).

Note: In laboratories with specialized "work cells" (a well defined group of analysts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented.

In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean quality system matrix (a sample of a quality system matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., drinking water, solids, biological tissue and air. However, before any results are reported using this method, actual sample spike results may be used to meet this standard, i.e., at least four consecutive matrix spikes within the last twelve months. In addition, for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples.

All demonstrations shall be documented through the use of the form in this appendix. All data applicable to the demonstration need not be attached to the form, but must be retained and available at the laboratory.

When an analyte not currently found on the laboratory's list of accredited analytes is added to an existing accredited test method, an initial evaluation must be performed for that analyte.

The following steps~~, which are adapted from the EPA test methods published in 40 CFR Part 136, Appendix A,~~ shall be performed if required by mandatory test method or regulation. ~~Note: For analytes for which spiking is not an option and for which quality control samples are not readily available, the 40 CFR approach is one way to perform this demonstration.~~ It is the responsibility of the laboratory to document that other approaches to DOC are adequate, this shall be documented in the laboratory's Quality Manual, e.g., for Whole Effluent Toxicity Testing see section D.2.1.a.1.

When laboratory clients provide the laboratory with Measurement Quality Objectives (MQO) that are inconsistent with or less stringent than the requirements of this standard, the client shall be notified in writing, prior to testing, of the difference between the client defined MQO and the applicable method or requirements of this standard.

a) A quality control sample shall be obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The analyte(s) shall be diluted in a volume of clean quality system matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 10 times the method-stated or laboratory-calculated method limit of detection ~~detection limit~~.

c) At least four aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using all of the results, calculate the mean recovery (0) (~~x~~)in the appropriate reporting units (such as g/L) and the standard deviations of the population sample (n-1) (in the same units) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory must assess performance against established and documented criteria.

e) Compare the information from (d) above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to 1) or 2) below.

   1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.

   2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, confirms a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

C.2 CERTIFICATION STATEMENT

The following certification statement shall be used to document the completion of each demonstration of capability. A copy of the certification statement shall be retained in the personnel records of each affected employee (see 5.5.2.5 and 5.4.12.2.5.4.b).

Demonstration of Capability
Certification Statement

Date: Page __of __
Laboratory Name:
Laboratory Address:
Analyst(s) Name(s):

Matrix:
(examples: laboratory pure water, soil, air, solid, biological tissue)
Method number, SOP#, Rev#, and Analyte, or Class of Analytes or Measured Parameters
(examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

_____	_____	_____
Technical Director's Name and Title	Signature	Date
_____	_____	_____
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data.

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.

Complete: Includes the results of all supporting performance testing.

Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

C.3 INITIAL TEST METHOD EVALUATION

For Chemistry, Radiochemistry, Air, and Asbestos Microscopy testing, initial test method evaluation requirements consist of the requirements specified in C.3. For Toxicity testing, and Microbiology testing, the initial test method evaluation requirements are contained at Appendix D.2. and D.3., respectively.

C.3.1. Limit of Detection (LOD)

The laboratory shall confirm the LOD for the method for each target analyte of concern in the relevant sample matrices.

All sample-processing steps of the analytical method shall be included in the determination of the LOD.

The validity of the LOD shall be confirmed by qualitative identification of the analyte(s) in a QC sample in each relevant quality system matrix containing the analyte at no more than 2-3X the LOD for single analyte tests and 1-4X the LOD for multiple analyte tests. This verification must be performed on every instrument that is to be used for analysis of samples and reporting of data.

A LOD study is not required for any component for which spiking solutions or quality control samples are not available such as temperature, or, when test results are not to be reported to the LOD (versus the limit of quantitation or working range of instrument calibration), according to Appendices D.1.2, D.4.5, D.5.4, and D.6.6. Where an LOD study is not determined, the laboratory may not report a value below the Limit of Quantitation.

C.3.2. Limit of Quantitation (LOQ)

The laboratory shall confirm the (LOQ) for each analyte of concern according to a defined, documented procedure, such as required in Appendix D.1.2.g.

The LOQ study is not required for any component or property for which spiking solutions or quality control samples are not commercially available or otherwise inappropriate (e.g., pH).

The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at or near the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria or client data quality objectives for accuracy (bias). This single analysis is not required if the bias and precision of the measurement system is evaluated at the LOQ.

C.3.3. Evaluation of Precision and Bias

The laboratory shall evaluate the Precision and Bias of a Standard Method for each analyte of concern for each quality system matrix according to the single-concentration four-replicate recovery study procedures in Appendix C.1 above (or alternate procedure documented in the quality manual when the analyte cannot be spiked into the sample matrix and QC samples are not commercially available).

For Laboratory-developed test methods or non-standard test methods as defined at 5.5.4.3 and 5.5.4.4. that were not in use by the laboratory before July 2003, the laboratory must have a documented procedure to evaluate precision and bias. The laboratory must also compare results of the precision and bias measurements with criteria established by the client, by criteria given in the reference method or criteria established by the laboratory.

Precision and bias measurements must evaluate the method across the analytical calibration range of the method. The laboratory must also evaluate precision and bias in the relevant quality system matrices and must process the samples through the entire measurement system for each analyte of interest.

Examples of a systematic approach to evaluate precision and bias could be the following:

> Analyze QC samples in triplicate containing the analytes of concern at or near the limit of quantitation, at the upper-range of the calibration (upper 20%) and at a mid-range concentration. Process these samples on different days as three sets of samples through the entire measurement system for each analyte of interest. Each day one QC sample at each concentration is analyzed. A separate method blank shall be subjected to the analytical method along with the QC samples on each of the three days. (Note that the three samples at the LOQ concentration can demonstrate sensitivity as well.) For each analyte, calculate the mean recovery for each day, for each level over days, and for all nine samples. Calculate the relative standard deviation for each of the separate means obtained. Compare the standard deviations for the different days and the standard deviations for the different concentrations. If the different standard deviations are all statistically insignificant (e.g., F-test), then compare the overall mean and standard deviation with the established criteria from above.
>
> A validation protocol such as the Tier I, Tier II, and Tier III requirements in US EPA Office of Water's Alternate Test Procedure (ATP) approval process.

C.3.4. Evaluation of Selectivity

The laboratory shall evaluate selectivity by following the checks established within the method, which may include mass spectral tuning, second column confirmation, ICP inter-element interference checks, chromatography retention time windows, sample blanks, spectrochemical absorption or fluorescence profiles, co-precipitation evaluations, and electrode response factors.

QUALITY SYSTEMS
APPENDIX D

ESSENTIAL QUALITY CONTROL REQUIREMENTS

Appendix D - ESSENTIAL QUALITY CONTROL REQUIREMENTS

The quality control protocols specified by the laboratory's method manual (5.5.4.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into their method manuals and/or the Laboratory Quality Manual.

All quality control measures shall be assessed and evaluated on an on-going basis and quality control acceptance criteria shall be used to determine the validity of the data. The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

The requirements from the body of Chapter 5, e.g., 5.5.9.2, apply to all types of testing. The specific manner in which they are implemented is detailed in each of the sections of this Appendix, i.e., chemical testing, W.E.T. testing, microbiology testing, radiochemical testing and air testing.

D.1 CHEMICAL TESTING

D.1.1 Positive and Negative Controls

D.1.1.1 Negative Control - Method Performance

a) Purpose: The method blank is used to assess the preparation batch for possible contamination during the preparation and processing steps. The method blank shall be processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure. Procedures shall be in place to determine if a method blank is contaminated. Any affected samples associated with a contaminated method blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.

b) Frequency: The method blank shall be analyzed at a minimum of 1 per preparation batch. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

c) Composition: The method blank shall consist of a quality system matrix that is similar to the associated samples and is known to be free of the analytes of interest.

d) Evaluation Criteria and Corrective Action: While the goal is to have no detectable contaminants, each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. The source of contamination shall be investigated and measures taken to minimize or eliminate the problem and affected samples reprocessed or data shall be appropriately qualified if:

   1) The concentration of a targeted analyte in the blank is at or above the reporting limit as established by the test method or by regulation, AND is greater than 1/10 of the amount measured in any sample.

   2) The blank contamination otherwise affects the sample results as per the test method requirements or the individual project data quality objectives.

3) When a blank is determined to be contaminated, the cause must be investigated and measures taken to minimize or eliminate the problem. Samples associated with a contaminated blank shall be evaluated as to the best corrective action for the samples (e.g. reprocessing or data qualifying codes). In all cases the corrective action must be documented.

D.1.1.2 Positive Control - Method Performance

D.1.1.2.1 Laboratory Control Sample (LCS)

a) Purpose: The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS are compared to established criteria and, if found to be outside of these criteria, indicates that the analytical system is "out of control". Any affected samples associated with an out of control LCS shall be reprocessed for re-analysis or the results reported with appropriate data qualifying codes.

b) Frequency: The LCS shall be analyzed at a minimum of 1 per preparation batch. Exceptions would be for those analytes for which no spiking solutions are available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

c) Composition: The LCS is a controlled quality system matrix, known to be free of analytes of interest, spiked with known and verified concentrations of analytes. NOTE: the matrix spike may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. Alternatively the LCS may consist of a media containing known and verified concentrations of analytes or as Certified Reference Material (CRM). All analyte concentrations shall be within the calibration range of the methods. The following shall be used in choosing components for the spike mixtures:

The components to be spiked shall be as specified by the mandated test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components the laboratory shall spike per the following:

For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected should be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2-year period.

1) For methods that include 1-10 targets, spike all components;

2) For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;

3) For methods with more than 20 targets, spike at least 16 components.

d) Evaluation Criteria and Corrective Action: The results of the individual batch LCS are calculated in percent recovery or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation.

The individual LCS is compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits or utilize client specified assessment criteria.

A LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be "out of control" shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.

e) If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. This may not indicate that the system is out of control, therefore corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit (3 standard deviations), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.

The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte lists with fewer than 11 analytes.

The number of allowable marginal exceedances is as follows:

1) >90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit;

2) 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit;

3) 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit;

4) 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit;

5) 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit;

6) <11 analytes in LCS, no analytes allowed in ME of the LCS control limit;

Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken. Laboratories must have a process to monitor the application of marginal exceedance allowance to the LCS to ensure random behavior.

D.1.1.3 Sample Specific Controls

The laboratory must document procedures for determining the effect of the sample matrix on method performance. These procedures relate to the analyses of quality system matrix specific Quality Control (QC) samples and are designed as data quality indicators for a specific sample using the designated test method. These controls alone are not used to judge laboratory performance.

Examples of matrix specific QC include: Matrix Spike (MS); Matrix Spike Duplicate (MSD); sample duplicates; and surrogate spikes. The laboratory shall have procedures in place for tracking, managing, and handling matrix specific QC criteria including spiking appropriate components at appropriate concentrations, calculating recoveries and relative percent difference, evaluating and reporting results based on performance of the QC samples.

D.1.1.3.1 Matrix Spike; Matrix Spike Duplicates

a) Purpose: Matrix specific QC samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. The information from these controls is sample/matrix specific and would not normally be used to determine the validity of the entire batch.

b) Frequency: The frequency of the analysis of matrix specific samples shall be determined as part of a systematic planning process (e.g. Data Quality Objectives) or as specified by the ~~required mandated~~ test method.

c) Composition: The components to be spiked shall be as specified by the mandated test method. Any permit specified analytes, as specified by regulation or client requested analytes shall also be included. If there are no specified components, the laboratory shall spike per the following:

For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test methods that have extremely long lists of analytes, a representative number may be chosen using the following criteria for choosing the number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2 year period.

1) For methods that include 1-10 targets, spike all components;

2) For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;

3) For methods with more than 20 targets, spike at least 16 components.

d) Evaluation Criteria and Corrective Action: The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given <u>quality system</u> matrix and are expressed as percent recovery (%R), relative percent difference (RPD), or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation for %R, RPD or other statistical treatment used.

The results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix spike results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

D.1.1.3.2 Matrix Duplicates

a) Purpose: Matrix duplicates are defined as replicate aliquots of the same sample taken through the entire analytical procedure. The results from this analysis indicate the precision of the results for the specific sample using the selected method. The matrix duplicate provides a usable measure of precision only when target analytes are found in the sample chosen for duplication.

b) Frequency: The frequency of the analysis of matrix duplicates may be determined as part of a systematic planning process (e.g. Data Quality Objectives) or as specified by the mandated test method.

c) Composition: Matrix duplicates are performed on replicate aliquots of actual samples. The composition is usually not known.

d) Evaluation Criteria and Corrective Action: The results from matrix duplicates are primarily designed to assess the precision of analytical results in a given quality system matrix and are expressed as relative percent difference (RPD) or another statistical treatment (e.g., absolute differences). The laboratory shall document the calculation for relative percent difference or other statistical treatments.

Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix duplicates results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

D.1.1.3.3 Surrogate Spikes

a) Purpose: Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistries of the targeted components of the method. Added prior to sample preparation/extraction, they provide a measure of recovery for every sample matrix.

b) Frequency: Except where the quality system matrix precludes its use or when not commercially available, surrogate compounds must be added to all samples, standards, and blanks for all appropriate test methods.

c) Composition: Surrogate compounds are chosen to represent the various chemistries of the target analytes in the method or MQO. They are often specified by the mandated method and are deliberately chosen for their being unlikely to occur as an environmental contaminant. Often this is accomplished by using deuterated analogs of select compounds.

d) Evaluation Criteria and Corrective Action: The results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory should determine internal criteria and document the method used to establish the limits. Surrogates outside the acceptance criteria must be evaluated for the effect indicated for the individual sample results. The appropriate corrective action may be guided by the data quality objectives or other site specific requirements. Results reported from analyses with surrogate recoveries outside the acceptance criteria should include appropriate data qualifiers.

D.1.2 Limit of Detection and Limit of Quantitation ~~Limits~~

All procedures used must be documented. Documentation must include the quality system matrix type. All supporting data must be retained.

D.1.2.1 Limit of Detection (LOD)

The laboratory shall utilize a test method that provides a LOD ~~detection limit~~ that is appropriate and relevant for the intended use of the data. An LOD is not required for a test method when test results are not reported outside of the calibration range. ~~Detection limits~~ LODs shall be determined by the protocol in the mandated test method or applicable regulation, e.g., Method Detection Limit (MDL) in accordance with C.3.1. If the protocol for determining LOD ~~detection limits~~ is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

a) ~~A detection limit study is not required for any component for which spiking solutions or quality control samples are not available such as temperature.~~

~~b)~~ The LOD ~~detection limit~~ shall be initially determined for the compounds of interest in each test method in a quality system matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the LOD ~~detection limit~~ must be determined in the quality system matrix of interest (see definition of matrix).

~~c~~b) LOD ~~Detection limits~~ must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

~~d) All sample processing steps of the analytical method shall be included in the determination of the detection limit.~~

~~e) All procedures used must be documented. Documentation must include the matrix type. All supporting data must be retained.~~

~~f~~c) The laboratory must have established procedures to relate LOD ~~detection limits~~ with LOQ ~~quantitation limits~~.

~~g~~d) ~~The test method's quantitation limits must be established and must be above the detection limits.~~The LOD must be verified annually for each quality system matrix, method and analyte according to the procedure specified in C.3.

D.1.2.2 Limit of Quantitation (LOQ)

a) Any established LOQ must be above the LOD

b) The LOQ must be verified annually for each quality system matrix, method and analyte according to the procedure specified in C.3. Alternatively, the annual LOQ verification is not required if the LOD is reevaluated or verified according to D.1.2.d above.

D.1.3 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.1.4 Quality of Standards and Reagents

a) The source of standards shall comply with 5.5.6.2.2.2.

b) Reagent Quality, Water Quality and Checks:

1) Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information shall be documented.

2) Water - The quality of water sources shall be monitored and documented and shall meet method specified requirements.

3) The laboratory will verify the concentration of titrants in accordance with written laboratory procedures.

D.1.5 Selectivity

a) ~~Absolute retention time and relative retention time aid in the identification of components in chromatographic analyses and to evaluate the effectiveness of a column to separate constituents. The laboratory shall develop and document acceptance criteria for retention time windows.~~ The laboratory shall evaluate selectivity by following the checks established within the method, which may include mass spectral tuning, second column confirmation, ICP inter-element interference checks, chromatography retention time windows, sample blanks, spectrochemical absorption or fluorescence profiles, co-precipitation evaluations, and electrode response factors.

b) A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.

c) The laboratory shall document acceptance criteria for mass spectral tuning.

D.1.6 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) Glassware Cleaning - Glassware shall be cleaned to meet the sensitivity of the test method.

Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.

D.2 TOXICITY TESTING

These standards apply to laboratories measuring the toxicity and/or bioaccumulation of contaminants in ~~general. They are applicable to toxicity or bioaccumulation test methods for evaluating e~~ffluents

(whole effluent toxicity or WET), receiving waters, sediments, elutriates, leachates and soils. In addition to the essential quality control standards described below, some methods may have additional or other requirements based on factors such as the type of quality system matrix evaluated. ~~Additional information can be found in the following methods manuals (or most recent edition): EPA/600/4-91/002, EPA/600/4-91/003, EPA/600/4-90/027F (WET testing), EPA/600/4-90/031 (general aquatic toxicity testing), EPA/600/R-94/025, EPA/600/R-94/024, EPA/503/R-91/001, EPA/823/B-98/004 (sediments and elutriates), EPA/600/3-88/029, EPA/600/3-89/013, ASTM E1598-94 and ASTM 1676-97 (soils).~~

D.2.1 Positive and Negative Controls

a) Positive Control - ~~Reference Toxicants -~~ Reference toxicant tests ~~indicate the sensitivity of the test organisms being used and~~ demonstrate a laboratory's ability to obtain consistent results with the test method and evaluate the overall health and sensitivity of test organisms over time.

1) The laboratory must demonstrate its ability to obtain consistent results with reference toxicants and complete an initial Demonstration of Capability (DOC) in order to attain accreditation in toxicity testing methods ~~before it performs toxicity tests with effluents or other environmental samples for regulatory compliance purposes~~.

i) An initial DOC shall consist of ~~To meet this requirement, the intra-laboratory precision must be determined by performing~~ five or more acceptable standard reference toxicant (SRT) tests for each test method and species with different batches of organisms. Appropriate ~~and appropriate~~ negative controls (water, sediment, or soil) shall be tested at the frequency and duration specified in the test method. Initial DOCs shall be prepared in accordance with the requirements of Appendix C.

ii) ~~An intralaboratory coefficient of variation (%CV) is not established for each test method. However, a testing laboratory shall maintain~~ Initial DOC is established by maintenance of SRT test results on control charts. A laboratory shall record ~~for~~ the control performance and ~~reference toxicant~~ statistical endpoints (such as NOEC or ECp) for each method on control charts. Initial DOC is established where the test results require in D.2.1 a) 1) i) fall within the control limits established in accordance with D.2.1 a) 1) iii) and meet test acceptability criteria (TAC). The laboratory shall evaluate precision (i.e. coefficient of variation, CV) or sensitivity (i.e. statistical minimum significant difference, (SMSD) measures (see D.2.1 a) 1) iv) for these tests against method specific or (lacking the former) laboratory-derived criteria to determine validity of the initial DOC. ~~and shall evaluate the intralaboratory variability with a specific reference toxicant for each test method.~~

iii) For endpoints that are point estimates (Icp, Ecp) control charts are constructed by plotting the cumulative mean and the control limits which consist of the upper and lower 95% confidence limits (+/- 2 standard deviations). In case of highly variable point estimates which exceed method-specific criteria the control chart limits are adjusted accordingly. For endpoints from hypothesis tests (NOEC, NOAEC) the values are plotted directly and the control limits consist of one concentration interval above and below the concentration representing the central tendency (i.e. the mode).

iv) For endpoints that are point estimates the cumulative mean CV is calculated and for endpoints from hypothesis tests, the SMSD is calculated. These values are maintained on a control chart.

2) Ongoing laboratory performance shall be demonstrated by routine SRT testing ~~performing regular reference toxicant tests~~ for each test method and species in accordance with the minimum frequency requirements specified in D.2.1.a.3.

i) Intralaboratory precision is determined on an ongoing basis through the use of control charts as established in D.2.1 a) 1) ii ~~must be determined through the use of reference toxicant tests and plotted in quality control charts.~~ The control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC50 for acute tests, or as appropriate hypothesis test values, such as the NOEC or NOAEC, over time within a laboratory.

ii) ~~For endpoints that are point estimates (ICp, ECp) control charts are constructed by plotting the cumulative mean and the control limits which consist of the upper and lower 95% confidence limits (+/- 2 std. dev.); these values are re-calculated with each successive test result. For endpoints from hypothesis tests (NOEC, NOAEC) the values are plotted directly and the control limits consist of one concentration interval above and below the concentration representing central tendency (i.e. the mode).~~

~~iii~~) After initial laboratory DOC is determined, the control limits and CV for an individual test method and species shall be adjusted as additional test results are obtained. After 20 data points are collected for a test method and species, the control chart is maintained using only the last 20 data points, i.e. each successive mean value and control limit is calculated using only the last 20 values.

~~iv~~iii) Control chart limits are expected to be exceeded occasionally regardless of how well a laboratory performs. Acceptance limits for point estimates (ICp, ECp) which are based on 95% confidence limits should theoretically be exceeded for one in twenty tests. Depending on the dilution factor and test sensitivity, control charts based on hypothesis test values (NOEC, NOAEC) may be expected to be exceeded on a similar frequency. Test results which fall outside of control chart limits at a frequency of 5% or less, or which fall just outside control chart limits (especially in the case of highly proficient laboratories which may develop relatively narrow acceptance limits over time), are not rejected *de facto.* Such data are evaluated in comparison with control chart characteristics including the width of the acceptance limits and the degree of departure of the value from acceptance limits.

~~v~~iv) Laboratories shall develop an acceptance/rejection policies, consistent with the test methods, ~~policy~~ for reference toxicant data which considers test dilution factor, test sensitivity (for hypothesis test values), testing frequency, out-of-control test frequency, relative width of acceptance limits, control chart CV, and degree of difference between test results and acceptance limits.

~~v~~iv) In the case of reference toxicant data which fails to meet control chart acceptance criteria~~, the results of environmental toxicity tests conducted during the affected period may be suspect and regarded as provisional. In this case~~ the test data are ~~procedure is~~ examined for defects, corrective action taken and the test repeated if necessary, using a different batch of organisms~~, as soon as possible~~ or the data is qualified.

3) The frequency of ongoing laboratory reference toxicant testing shall be as follows unless the method specifically requires less frequent SRT tests (e.g. sediment tests) ~~comply with the EPA or state permitting authority requirements. The following minimum frequency shall be met~~:

~~i) Each batch of test organisms obtained from an outside source, field collection or from laboratory spawning of field-collected species not amenable to routine laboratory culture (for example, sea urchins and bivalve mollusks) must be evaluated with a reference toxicant test of the same type as the environmental toxicity test within the seven days preceding the test or concurrently with the test.~~

~~ii) Test organisms obtained from in-house laboratory cultures must be tested with reference toxicant tests at least once each month for each test method. However, if a given species produced by in-house cultures is used only monthly, or less frequently, a reference toxicant test of the same type must be performed with each environmental toxicity test.~~

~~iii) For test methods and species commonly used in the laboratory, but which are tested on a seasonal basis (e.g. sea urchin fertilization tests), reference toxicant tests must be conducted for each month the method is in use.~~

i) For test methods conducted at a frequency of greater then monthly, SRT tests shall be conducted at an ongoing frequency of monthly.

ii) For test methods and species commonly used in the laboratory, but which are tested at a frequency of monthly or less, SRT tests shall be conducted concurrently with the environmental test.

iii) If the test organisms are obtained from an outside source the sensitivity of each batch of organisms received from a supplier shall be determined via a concurrent SRT test unless the supplier can provide control chart data for the last five SRT tests using the same SRT and test conditions employed by the laboratory.

iv) The DOC for an analyst shall be consistent with 5.5.2.6.c)3) but the frequency need not exceed the method specified requirements and D.2.1 a) 3).

4) These standards do not currently specify a particular reference toxicant and dilution series however, if the state or permitting authority identifies a reference toxicant or dilution series for a particular test, the laboratory shall follow the specified requirements. All reference toxicant tests conducted for a given test method and species must use the same reference toxicant, test concentrations, dilution water and data analysis methods. A dilution factor of 0.5x or greater shall be used for both acute and chronic tests.

5) The reference toxicant tests shall be conducted following the same procedures as the environmental toxicity tests for which the precision is being evaluated~~.~~, unless otherwise specified in the test method (for example, 10-day sediment tests employ 96-h water-only reference toxicant tests). The test duration, laboratory dilution ~~or control~~ water, feeding, organism age, ~~age~~ range and density, test volumes, renewal frequency, water quality measurements, and the number of test concentrations, replicates and organisms per replicate shall be the same as specified for the environmental toxicity test.

6) If several variations of a test method are used by the laboratory (e.g. 48-hour static acute, 48-hour renewal acute, 96-hour renewal acute) the reference test for the method shall be based on the variation with the longest exposure or greatest degree of laboratory manipulations.

b) Negative Control - Control, Brine Control, Control Sediment, Control Soil or Dilution Water -

1) The standards for the use, type and frequency of testing of negative controls are specified by the test methods and by permit or regulation and shall be followed. A negative control is included with each test to evaluate test performance and the health and sensitivity of the specific batch of organisms.

2) Appropriate additional negative controls shall be included when sample adjustments (for example addition of ~~sodium hydroxide for pH adjustment or~~ thiosulfate for dechlorination) or solvent carriers are used in the test.

3) Test Acceptability Criteria (TAC) - The test acceptability criteria ~~(for example, the whole-effluent chronic *Ceriodaphnia* test, requires 80% or greater survival and an average 15 young per female in the controls) as~~ specified in the test method must be achieved for both the reference toxicant and the effluent or environmental sample toxicity test. The criteria shall be calculated and shall meet the method specified requirements for performing toxicity tests.

D.2.2 Variability and/or Reproducibility

Intralaboratory precision shall be determined on an ongoing basis through the use of further reference toxicant tests and related control charts as described in item D.2.1.a above.

D.2.3 Accuracy

This principle is not applicable to Toxicity Testing.

D.2.4 Test Sensitivity

a) The ~~If the Dunnett's procedure is used, the statistical minimum significant difference~~ (SMSD) shall be calculated according to the formula specified by the test method and reported with the test results.

b) ~~Estimate the SMSD for non-normal distribution and or heterogenous variances.~~

~~c)~~ Point estimates: (LCp, ICp, or ECp) - Confidence intervals shall be reported as a measure of the precision around the point estimate value, when the calculation is possible.

~~d~~c) The SMSD shall be calculated and reported for only hypothesis test values, such as the NOEC or NOAEC.

D.2.5 Selection of Appropriate Statistical Analysis Methods

a) If required, methods of data analysis and endpoints are specified by language in the regulation, permit or the test method.

b) Dose Response Curves – The ~~When required, the~~ data shall be plotted in the form of a curve relating the dose of the chemical or concentration of sample to cumulative percentage of test

organisms demonstrating a response such as death. Evaluation criteria shall be established for interpretation of concentration or dose response curves.

D.2.6 Selection and Use of Reagents and Standards

a) The grade of all reagents used in toxicity tests is specified in the test method except the reference standard. All reference standards shall be prepared from chemicals which are analytical reagent grade or better. The preparation of all standards and reference toxicants shall be documented.

b) All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH or specific conductance, shall comply with the standards outlined in 5.5.5.2.1.d ~~Section 5.5.5.2 above~~.

c) Only reagent-grade water collected from distillation or deionization units ~~(> 17 megohm resistivity)~~ is used to prepare reagents.

D.2.7 Selectivity

This principle is not applicable. The selectivity of the test is specified by permit or regulation.

D.2.8 Constant and Consistent Test Conditions

a) If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid ~~loss of cultures due to~~ cross-contamination.

b) Laboratory space must be adequate for the types and numbers of tests performed. The building must provide adequate cooling, heating and illumination for conducting testing and culturing; hot and cold running water must be available for cleaning equipment.

c) Air used for aeration of test solutions, dilution waters and cultures must be free of oil and fumes.

d) The laboratory or a contracted outside expert shall positively identify test organisms to species on an annual basis. The taxonomic reference (citation and page(s)) and the names(s) of the taxonomic expert(s) must be kept on file at the laboratory. When organisms are obtained from an outside source the supplier must provide this same information.

e) Instruments used for routine support measurements of chemical and physical parameters such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, ammonia and weight shall be calibrated, and/or standardized per manufacturer's instructions. As these are support measurements, only the calibration and verification requirements specified at 5.5.5.2.1 apply. ~~and Section 5.5.5.2. Temperature shall be calibrated per section 5.5.5.2.1.~~ All measurements and calibrations shall be documented.

f) Test temperature shall be maintained as specified for the test method. Temperature control equipment must be adequate to maintain the required test temperature(s). The average daily temperature of the test solutions must be maintained within ~~1°C of~~ the method specified range ~~selected test temperature, for the duration of the test~~. The minimum frequency of measurement shall be once per 24 hour period. The test temperature for continuous-flow toxicity tests shall be recorded and monitored continuously. Where electronic data loggers are used, temperature shall be monitored at a frequency sufficient to capture temporal variations of the environmental control system.

g) Reagent grade water, prepared by any combination of distillation, reverse osmosis, ion exchange, activated carbon and particle filtration, shall meet the method specified requirements. ~~following requirements as verified by monthly measurement: conductivity less than or equal to 0.1 mho/cm or resistivity greater than or equal to 17 megohms, pH 5.5 to 7.5 S.U. and total residual chlorine non-detectable. (1 mho/cm = 1 S/cm)~~

h) The quality of the standard dilution water used for testing or culturing must be sufficient to allow satisfactory survival, growth and reproduction of the test species as demonstrated by routine reference toxicant tests and negative control performance. Water used for culturing and testing shall be analyzed for toxic metals and organics whenever the minimum acceptability criteria for control survival, growth or reproduction are not met and no other cause, such as contaminated glassware or poor stock, can be identified. It is recognized that the analyte lists of some methods manuals may not include all potential toxicants, are based on estimates of chemical toxicity available at the time of publication and may specify detection limits which are not achievable in all matrices. However, for those analytes not listed, or for which the measured concentration or limit of detection ~~detection limit~~ is greater than the method-specified limit, the laboratory must demonstrate that the analyte at the measured concentration or reported limit of detection ~~detection limit~~ does not exceed one tenth the expected chronic value for the most sensitive species tested and/or cultured. The expected chronic value is based on professional judgment and the best available scientific data. The "USEPA Ambient Water Quality Criteria Documents" and the EPA AQUIRE data base provide guidance and data on acceptability and toxicity of individual metals and organic compounds.

i) The quality of the food used for testing or culturing must be sufficient to allow satisfactory survival, growth and reproduction of the test species as demonstrated by routine reference toxicant tests and negative control performance. ~~For each new batch of laboratory-prepared or lot of commercial food used by the laboratory, the performance of organisms fed with the new food shall be compared with the performance of organisms fed with a food of known quality. If the food is used for culturing, its suitability is determined using a measure that evaluates the effect of food quality on survival and growth or reproduction of each of the relevant test species. Where applicable, foods used only in chronic toxicity tests are evaluated using the reference toxicant regularly employed in the laboratory QA program and compared with results of previous test(s) using a food of known quality. In the case of algae, rotifers or other cultured foods, which are collected as a continuous batch, the quality is assessed as described above, each time new nutrient stocks are prepared, a new starter culture is employed or when a significant change in culture conditions occurs.~~ The laboratory shall have written procedures for the ~~statistical~~ evaluation of food acceptance.

j) Organisms ~~Food used to culture organisms~~ used in bioaccumulation tests must be analyzed at the start of the test (baseline) for the target compounds to be measured in the bioaccumulation tests.

k) Test chamber size and test solution volume shall be as specified in the test method. All test chambers used in a test must be identical.

l) Test organisms shall be fed the quantity and type food or nutrients specified in the test method. They shall also be fed at the intervals specified in the test methods.

m) All organisms in a test must be from the same source. Where available certified seeds are used for soil tests.

n) All organisms used in tests, or used as broodstock to produce neonate test organisms (for example cladocerans and larval fish), must appear healthy, show no signs of stress or disease and exhibit acceptable survival (90% or greater) during the 24 hour period immediately preceding use in tests.

o) All materials used for test chambers, culture tanks, tubing, etc. and coming in contact with test samples, solutions, control water, sediment or soil or food must be non-toxic and cleaned as described in the test methods. Materials must not reduce or add to sample toxicity. Appropriate materials for use in toxicity testing and culturing are described in the referenced manuals.

p) Light intensity shall be maintained as specified in the methods manuals. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the test methods and shall be documented at least quarterly. For algal and plant tests, the light intensity shall be measured and recorded at the start of each test.

~~q) At a minimum, during aquatic chronic testing DO and pH shall be measured daily in at least one replicate of each concentration. In static-renewal tests DO must be measured at both the beginning and end of each 24-h exposure period and may be measured in old and new solutions prior to organism transfer, or after organism transfer; pH is measured at the end of each exposure period (i.e. in old solutions).~~

~~r)~~ The health and culturing conditions of all organisms used for testing shall be documented by the testing laboratory. Such documentation shall include culture conditions (e.g. salinity, hardness, temperature, pH) and observations of any stress, disease or mortality. When organisms are obtained from an outside source, the laboratory shall obtain written documentation of these water quality parameters and biological observations for each lot of organism received. These observations shall adequately address the 24-hour time period referenced in item D.2.8.n. above. The laboratory shall also record each of these observations and water quality parameters upon the arrival of the organisms at the testing laboratory.

~~s~~r) Age and the age range of the test organisms must be as specified in the test method. Supporting information, such as hatch dates and times, times of brood releases and metrics (for example, chironomid head capsule width) shall be documented.

~~t~~s) The maximum holding time of effluents (elapsed time from sample collection to first use in a test) shall not exceed 36 hours; samples may be used for renewal up to 72 hours after first use. ~~and the last use of the sample in test renewals shall not exceed 72 hours without the permission of the permitting authority.~~

~~u~~t) All samples shall be chilled to 0 to 6 ~~4~~°C during or immediately after collection (see requirements in section 5.5.8.3.1).

~~v~~u) Organisms used in a given test ~~obtained from an outside source~~ must be from the same batch. Chronic tests shall have a minimum of four replicates per treatment.

v) Chronic tests shall have a minimum of four replicates per treatment.

w) The control population of Ceriodaphnia in chronic effluent or receiving water tests shall contain no more than 20% males.

x) The culturing of C. dubia shall be adequate such that blocking by parentage can be established.

~~x~~y) Dissolved oxygen and pH in aquatic tests shall be within acceptable range at test initiation and aeration (minimal) is provided to tests if, and only if, acceptable dissolved oxygen concentrations cannot be otherwise maintained or if specified by the test method.

z) Test ~~The test~~ soils or sediments must be within the geochemical tolerance range of the test organism.

aa) An individual test may be conditionally acceptable if temperature, dissolved oxygen, pH and other specified conditions fall outside specifications, depending on the degree of the departure and the objectives of the tests (see test conditions and test acceptability criteria specified for each test method). The acceptability of the test shall depend on the experience and professional judgment of the technical director ~~employee~~ and the permitting authority.

D.3 MICROBIOLOGY TESTING

These standards apply to laboratories undertaking microbiological analysis of environmental samples. Microbiological testing refers to and includes the detection, isolation, enumeration, or identification of microorganisms and/or their metabolites, or determination of the presence or absence of growth in materials and media.

D.3.1 Sterility Checks and Blanks, Positive and Negative Controls

a) Sterility Checks and Blanks

The laboratory shall demonstrate that the filtration equipment and filters, sample containers, media and reagents have not been contaminated through improper handling or preparation, inadequate sterilization, or environmental exposure.

1) A sterility blank shall be analyzed for each lot of pre-prepared, ready-to-use medium (including chromofluorogenic reagent) and for each batch of medium prepared in the laboratory. This shall be done prior to first use of the medium.

2) For filtration technique, the laboratory shall conduct one beginning and one ending sterility check for each laboratory sterilized filtration unit used in a filtration series. The filtration series may include single or multiple filtration units, which have been sterilized prior to beginning the series. For pre-sterilized single use funnels a sterility check shall be performed on one funnel per lot. The filtration series is considered ended when more than 30 minutes elapses between successive filtrations. During a filtration series, filter funnels must be rinsed with three 20-30 ml portions of sterile rinse water after each sample filtration. In addition, laboratories must insert a sterility blank after every 10 samples or sanitize filtration units by UV light after each sample filtration.

3) For pour plate technique, sterility blanks of the medium shall be made by pouring, at a minimum, one uninoculated plate for each lot of pre-prepared, ready-to-use media and for each batch of medium prepared in the laboratory.

4) Sterility checks on sample containers shall be performed on at least one container for each lot of purchased, pre-sterilized containers. For containers prepared and sterilized in the laboratory, a sterility check shall be performed on one container per sterilized batch with non-selective growth media.

5) A sterility blank shall be performed on each batch of dilution water prepared in the laboratory and on each batch of pre-prepared, ready-to-use dilution water with non-selective growth media.

6) At least one filter from each new lot of membrane filters shall be checked for sterility with non-selective growth media.

b) Positive Controls

Positive culture controls demonstrate that the medium can support the growth of the target organism(s), and that the medium produces the specified or expected reaction to the target organism(s).

1) Each pre-prepared, ready-to-use lot of medium (including chromofluorogenic reagent) and each batch of medium prepared in the laboratory shall be tested with at least one pure culture of a known positive reaction. This shall be done prior to first use of the medium.

c) Negative Controls

Negative culture controls demonstrate that the medium does not support the growth of non-target organisms or does not demonstrate the typical positive reaction of the target organism(s).

Each pre-prepared, ready-to-use lot of selective medium (including chromofluorogenic reagent) and each batch of selective medium prepared in the laboratory shall be analyzed with one or more known negative culture controls, i.e. non-target organisms, as appropriate to the method. This shall be done prior to first use of the medium.

D.3.2 Test Variability/Reproducibility

For test methods that specify colony counts such as membrane filter or plated media, duplicate counts shall be performed monthly on one positive sample, for each month that the test is performed. If the lab has two or more analysts, each analyst shall count typical colonies on the same plate. Counts must be within 10% difference to be acceptable. In a laboratory with only one microbiology analyst, the same plate shall be counted twice by the analyst, with no more than 5% difference between the counts.

D.3.3 Method Evaluation

a) Laboratories are required to demonstrate proficiency with the test method prior to first use. This shall be achieved by comparison to a method already approved for use in the laboratory, or by analyzing a minimum of ten spiked samples whose quality system matrix is representative of those normally submitted to the laboratory, or by analyzing and passing one proficiency test series
provided by an approved proficiency sample provider. The laboratory shall maintain this documentation as long as the method is in use and for at least 5 years past the date of last use.

b) Laboratories shall participate in the Proficiency Test programs identified by NELAP (5.4.1.5.k or 5.5.9.1). The results of these analyses shall be used to evaluate the ability of the laboratory to produce acceptable data.

D.3.4 Test Performance

a) All growth and recovery media must be checked to assure that the target organism(s) respond in an acceptable and predictable manner (see D.3.1.b).

b) To ensure that analysis results are accurate, target organism identity shall be verified as specified in the method, e.g. by use of the completed test, or by use of secondary verification tests such as a catalase test.

D.3.5 Data Reduction

The calculations, data reduction and statistical interpretations specified by each test method shall be followed.

D.3.6 Quality of Standards, Reagents and Media

The laboratory shall ensure that the quality of the reagents and media used is appropriate for the test concerned.

a) Culture media may be prepared from commercial dehydrated powders or may be purchased ready to use. Media may be prepared by the laboratory from basic ingredients when commercial media are not available or when it can be demonstrated that commercial media do not provide adequate results. Media prepared by the laboratory from basic ingredients must be tested for performance (e.g., for selectivity, sensitivity, sterility, growth promotion, growth inhibition) prior to first use. Detailed testing criteria information must be defined in either the laboratory's test methods, SOPs, Quality Manual, or similar documentation.

b) Reagents, commercial dehydrated powders and media shall be used within the shelf-life of the product and shall be documented according to 5.5.6.4.

c) Distilled water, deionized water or reverse-osmosis produced water free from bactericidal and inhibitory substances shall be used in the preparation of media, solutions and buffers. The quality of the water shall be monitored for chlorine residual, specific conductance, and heterotrophic bacteria plate count monthly (when in use), when maintenance is performed on the water treatment system, or at startup after a period of disuse longer than one month.

   Analysis for metals and the Bacteriological Water Quality Test (to determine presence of toxic agents or growth promoting substances) shall be performed annually. Results of these analyses shall meet the specifications of the required method and records of analyses shall be maintained for five years. (An exception to performing the Bacteriological Water Quality Test shall be given to laboratories that can supply documentation to show that their water source meets the criteria, as specified by the method, for Type I or Type II reagent water.)

d) Media, solutions and reagents shall be prepared, used and stored according to a documented procedure following the manufacturer's instructions or the test method. Documentation for media prepared in the laboratory shall include date of preparation, preparer's initials, type and amount of media prepared, manufacturer and lot number, final pH of the media, and expiration date. Documentation for media purchased pre-prepared, ready-to-use shall include manufacturer, lot number, type and amount of media received, date of receipt, expiration date of the media, and pH of the media.

D.3.7 Selectivity

a) In order to ensure identity and traceability, reference cultures used for positive and negative controls shall be obtained from a recognized national collection, organization, or manufacturer recognized by the NELAP Accrediting Authority. Microorganisms may be single use preparations or cultures maintained by documented procedures that demonstrate the continued purity and viability of the organism.

   1) Reference cultures may be revived (if freeze-dried) or transferred from slants and subcultured once to provide reference stocks. The reference stocks shall be preserved by a technique which maintains the characteristics of the strains. Reference stocks shall be used to prepare working stocks for routine work. If reference stocks have been thawed, they must not be re-frozen and re-used.

   2) Working stocks shall not be sequentially cultured more than five times and shall not be subcultured to replace reference stocks.

D.3.8 Constant and Consistent Test Conditions

a) Laboratory Facilities

   Floors and work surfaces shall be non-absorbent and easy to clean and disinfect. Work surfaces shall be adequately sealed. Laboratories shall provide sufficient storage space, and shall be clean and free from dust accumulation. Plants, food, and drink shall be prohibited from the laboratory work area.

b) Laboratory Equipment

   1) Temperature Measuring Devices

      Temperature measuring devices such as liquid-in-glass thermometers, thermocouples, and platinum resistance thermometers used in incubators, autoclaves and other equipment shall be the appropriate quality to meet specification(s) in the test method. The graduation of the temperature measuring devices must be appropriate for the required accuracy of measurement and they shall be calibrated to national or international standards for temperature (see 5.5.6.2.2.2). Calibration shall be done at least annually.

   2) Autoclaves

      i) The performance of each autoclave shall be initially evaluated by establishing its functional properties and performance, for example heat distribution characteristics with respect to typical uses. Autoclaves shall meet specified temperature tolerances. Pressure cookers shall not be used for sterilization of growth media.

      ii) Demonstration of sterilization temperature shall be provided by use of continuous temperature recording device or by use of a maximum registering thermometer with every cycle. Appropriate biological indicators shall be used once per month to determine effective sterilization. Temperature sensitive tape shall be used with the contents of each autoclave run to indicate that the autoclave contents have been processed.

      iii) Records of autoclave operations shall be maintained for every cycle. Records shall include: date, contents, maximum temperature reached, pressure, time in sterilization mode, total run time (may be recorded as time in and time out) and analyst's initials.

iv) Autoclave maintenance, either internally or by service contract, shall be performed annually and shall include a pressure check and calibration of temperature device. Records of the maintenance shall be maintained in equipment logs.

v) The autoclave mechanical timing device shall be checked quarterly against a stopwatch and the actual time elapsed documented.

3) Volumetric Equipment

Volumetric equipment shall be calibrated as follows:

i) equipment with movable parts such as automatic dispensers, dispensers/diluters, and mechanical hand pipettes shall be calibrated quarterly.
ii) equipment such as filter funnels, bottles, non-class A glassware, and other marked containers shall be calibrated once per lot prior to first use.
iii) the volume of the disposable volumetric equipment such as sample bottles, disposable pipettes, and micropippette tips shall be checked once per lot.

4) UV Instruments

UV instruments, used for sanitization, shall be tested quarterly for effectiveness with an appropriate UV light meter or by plate count agar spread plates. Replace bulbs if output is less than 70% of original for light tests or if count reduction is less than 99% for a plate containing 200 to 300 organisms.

5) Conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments shall be calibrated according to the method specified requirements (see Section 5.5.5.2.1.d).

6) Incubators, Water Baths, Ovens

i) The ~~stability and~~ uniformity of temperature distribution ~~and time required after test sample addition to re-establish equilibrium conditions~~ in incubators and water baths shall be established. Temperature of incubators and water baths shall be documented twice daily, at least four hours apart, on each day of use.

ii) Ovens used for sterilization shall be checked for sterilization effectiveness monthly with appropriate biological indicators. Records shall be maintained for each cycle that include date, cycle time, temperature, contents and analyst's initials.

7) Labware (Glassware and Plasticware)

i) The laboratory shall have a documented procedure for washing labware, if applicable. Detergents designed for laboratory use must be used.

ii) Glassware shall be made of borosilicate or other non-corrosive material, free of chips and cracks, and shall have readable measurement marks.

iii) Labware that is washed and reused shall be tested for possible presence of residues which may inhibit or promote growth of microorganisms by performing the Inhibitory Residue Test annually, and each time the lab changes the lot of detergent or washing procedures.

iv) Washed labware shall be tested at least once daily, each day of washing, for possible acid or alkaline residue by testing at least one piece of labware with a suitable pH indicator such as bromothymol blue. Records of tests shall be maintained.

D.4 RADIOCHEMICAL TESTING

These standards apply to laboratories undertaking the examination of environmental samples by radiochemical analysis. These procedures for radiochemical analysis may involve some form of chemical separation followed by detection of the radioactive decay of analyte (or indicative daughters) and tracer isotopes where used. For the purpose of these standards procedures for the determination of radioactive isotopes by mass spectrometry (e.g. ICP-MS or TIMS) or optical (e.g. KPA) techniques are not addressed herein.

D.4.1 Negative and Positive Controls

a) Negative Controls

1) Method Blank - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess the batch. The method blank result shall be assessed against the specific acceptance criteria [see 5.5.4.1.2.b)18] specified in the laboratory method manual [see 5.5.4.1.2]. When the specified method blank acceptance criteria is not met the specified corrective action and contingencies [see 5.5.4.1.2.b) 19 and 20] shall be followed and results reported with appropriate data qualifying codes. The occurrence of a failed method blank acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.5.10.3.1.a].

2) In the case of gamma spectrometry which is a non-destructive analysis, ~~where the sample matrix is simply aliquoted into a calibrated counting geometry the~~ method blank shall be prepared using a calibrated ~~of similar~~ counting geometry ~~that is~~ similar to that used for the samples and the container of the appropriate geometry can be empty or filled to similar volume ~~with ASTM Type II water~~ to partially simulate gamma attenuation due to a sample matrix.

3) There shall be no subtraction of the required method blank [see D.4.1.a)1] result from the sample results in the associated preparation or analytical batch unless permitted by method or program. This does not preclude the application of any correction factor (e.g. instrument background, analyte presence in tracer, reagent impurities, peak overlap, ~~calibration blank,~~ etc.) to all analyzed samples, both program/project submitted and internal quality control samples. However, these correction factors shall not depend on the required method blank result in the associated analytical batch.

4) The method blank sample shall be prepared with similar aliquot size to that of the routine samples for analysis and the method blank result and acceptance criteria [5.5.4.1.2.b)18] shall be calculated in a manner that compensates for sample results based upon differing aliquot size.

b) Positive Controls

1) Laboratory Control Samples - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess the batch. The laboratory control sample result shall be assessed against the specific acceptance criteria [see 5.5.4.1.2.b)18] specified in the laboratory method manual [see 5.5.4.1.2]. When the specified laboratory control sample acceptance criteria is not met the specified corrective action and contingencies [see 5.5.4.1.2.b)19 and 20] shall be followed. The occurrence of a failed laboratory control sample acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.5.10.3.1.a].

2) Matrix Spike - Shall be performed at a frequency of one per preparation batch for those methods which include a chemical separation process without the use of an internal standard or carrier, and where there is sufficient sample to do so. Although gross alpha, gross beta and tritium measurements so not involve a chemical separation process, matrix spikes shall be performed for these analysis on aqueous samples ~~do not utilize an internal standard or carrier, for which there is a chemical separation process, and where there is sufficient sample to do so. The exceptions are gross alpha, gross beta and tritium which shall require matrix spikes for aqueous samples~~. The results of this analysis shall be one of the quality control measures to be used to assess the batch . The matrix spike result shall be assessed against the specific acceptance criteria [see 5.5.4.1.2.b)18] specified in the laboratory method manual [see 5.5.4.1.2]. When the specified matrix spike acceptance criteria is not met, the specified corrective action and contingencies [see 5.5.4.1.2.b)19 and 20] shall be followed. The occurrence of a failed matrix spike acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.5.10.3.1.a]. The lack of sufficient sample aliquot size to perform a matrix spike shall be noted in the laboratory report.

3) The activity of the laboratory control sample shall: (1) be at least 5 times ~~two to ten times~~ the limit of detection ~~detection limit or~~ and (2) at a level comparable to that of routine samples if the sample activities are expected to exceed 5 ~~10~~ times the limit of detection ~~detection limit~~.

4) The activity of the matrix spike analytes(s) shall be greater than five ~~ten~~ times the limit of detection ~~detection limit~~.

5) The laboratory standards used to prepare the laboratory control sample and matrix spike shall be from a source independent of the laboratory standards used for instrument calibration and must meet the requirements for reference standards provided in D.4.7 a).

6) The matrix spike shall be prepared by adding a known activity of target analyte after subsampling if required but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.). Where a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope (e.g. plutonium, Pu 238 and Pu 239, using alpha spectrometry), only one of the analyte isotopes need be included in the laboratory control or matrix spike sample at the indicated activity level. However, where more than one analyte isotope is present above the specified limit of detection ~~detection limit~~ each shall be assessed against the specified acceptance criteria.

7) Where gamma spectrometry is used to identify and quantitate more than one analyte isotope the laboratory control sample ~~and matrix spike~~ shall contain isotopes that represent the low (e.g. americium-241), medium (e.g. cesium-137) and high (e.g. cobalt-60) energy range of the analyzed gamma spectra. As indicated by these examples the

isotopes need not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated.

8) The laboratory control sample shall be prepared with similar aliquot size to that of the routine samples for analyses.

c) Other Controls

1) Tracer - For those methods that utilize a tracer (i.e. internal standard) each sample result shall have an associated tracer recovery calculated and reported. The tracer shall be added to the sample after subsampling if required but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method. The tracer recovery for each sample result shall be one of the quality control measures to be used to assess the associated sample result acceptance. The tracer recovery shall be assessed against the specific acceptance criteria [see 5.5.4.1.2.b)18] specified in the laboratory method manual [see 5.5.4.1.2]. When the specified tracer recovery acceptance criteria is not met the specified corrective action and contingencies [see 5.5.4.1.2.b)19 and 20] shall be followed. The occurrence of a failed tracer recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.5.10.3.1.a].

2) Carrier - For those methods that utilize a carrier for recovery determination, each sample shall have an associated carrier recovery calculated and reported. The carrier shall be added to the sample after subsampling if required but before any chemical treatment (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method. The carrier recovery for each sample shall be one of the quality control measures to be used to assess the associated sample result acceptance. The carrier recovery shall be assessed against the specific acceptance criteria [see 5.5.4.1.2.b)18] specified in the laboratory method manual [see 5.5.4.1.2]. When the specified carrier recovery acceptance criteria is not met the specified corrective action and contingencies [see 5.5.4.1.2.b)19 and 20] shall be followed. The occurrence of a failed carrier recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.5.10.3.1.a].

D.4.2 Analytical Variability/Reproducibility

a) Replicate - Shall be performed at a frequency of one per preparation batch where there is sufficient sample to do so. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The replicate result shall be assessed against the specific acceptance criteria [see 5.5.4.1.2.b)18] specified in the laboratory method manual [see 5.5.4.1.2]. When the specified replicate acceptance criteria is not met the specified corrective action and contingencies [see 5.5.4.1.2.b)19 and 20] shall be followed. ~~The corrective action shall consider the fact that sample inhomogeneity may be a cause of the failed replicate acceptance criteria.~~ The occurrence of a failed replicate acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.5.10.3.1.a].
b) For low level samples (less than approximately three times the limit of detection ~~detection limit~~) the laboratory may analyze duplicate laboratory control samples or a replicate matrix spike (matrix spike and a matrix spike duplicate) to determine reproducibility within a preparation batch.

D.4.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Initial Demonstration of Capability - (section 5.5.4.2.2 and Appendix C) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type (e.g., different detection technique), personnel or method.

b) Proficiency Test Samples - The results of such analysis (5.4.1.5.k and 5.5.9.1) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.4.4 Radiation Measurement Instrumentation ~~System Calibration~~

Because of the stability and response nature of modern radiation measurement instrumentation, it is not typically necessary to verify calibrate of these systems each day of use. However, verification of calibration is required as outlined in (b) below. This section addresses those practices that are necessary for proper calibration and those requirements of section 5.5.5.2.2 (Instrument Calibrations) that are not applicable to some types of radiation measurement instrumentation.

a) ~~Initial~~ Instrument Calibration

1) Given that activity detection efficiency is independent of sample activity at all but extreme activity levels, the requirements of subsections f, h and i of 5.5.5.2.2.1 are not applicable to radiochemical method calibrations except mass attenuation in gas-proportional counting and sample quench in liquid scintillation counting~~ Radiochemistry analytical instruments are subject to calibration when purchased, when the instrument is serviced, when the instrument is moved and when the instrument setting(s) have been changed~~. Radiation measurement instruments are subject to calibration prior to initial use, when the instrument is placed back in service after malfunctioning and the instrument's response has changed as determined by a performance check or when the instrument's response exceeds predetermined acceptance criteria for the instrument quality control.

2) Instrument calibration shall be performed with reference standards as defined in section D.4.7a. The standards shall have the same general characteristics (i.e., geometry, homogeneity, density, etc.) as the associated samples.

3) The frequency of calibration shall be addressed in the laboratory method manual [see 5.5.4.1.2.b)13] if not specified ~~addressed~~ in the method. A specific frequency (e.g. monthly) or observations from the associated control or tolerance chart, as the basis for calibration shall be specified.

b) Continuing Instrument Calibration Verification (Performance Checks)

~~Calibration verification~~Performance checks shall be performed using appropriate check sources and monitored with control charts or tolerance charts to ensure that the instrument is operating properly and that the detector response has not significantly changed and therefore the instrument calibration has not changed~~calibration has not changed~~. The same check source used in the preparation of the tolerance chart or control chart at the time of calibration shall be used in the calibration verification of the instrument. The check sources must provide adequate counting statistics for a relatively short count time and the source should be sealed or encapsulated to prevent loss of activity and contamination of the instrument and laboratory personnel.~~ For alpha and gamma spectroscopy systems, the instrument calibration verification shall include checks on the counting efficiency and the relationship between channel number and alpha or gamma ray energy.~~

1) For gamma spectroscopy systems, the performance ~~calibration verification~~ checks for efficiency and energy calibration shall be performed on a day of use basis along with performance checks on peak resolution.

2) For alpha spectroscopy systems, the performance ~~calibration verification~~ check for energy calibration shall be performed on a weekly basis and the performance check for counting efficiency shall be performed on at least a monthly basis.

3) For gas-proportional and liquid scintillation counters, the performance ~~calibration verification~~ check for counting efficiency shall be performed on a day of use basis. For batches of samples that uninterruptedly count for more than a day a performance check can be performed at the beginning and end of the batch as long as this time interval is no greater than one week. Verification of instrument calibration does not directly verify secondary calibrations, e.g., the mass efficiency curve or the quench curve.

4) For scintillation counters the calibration verification for counting efficiency shall be performed on a day of use basis.

c) Background Measurement

Background measurements shall be made on a regular basis and monitored using control charts or tolerance charts to ensure that a laboratory maintains its capability to meet required data quality objectives. These values may be ~~are~~ subtracted from the total measured activity in the determination of the sample activity.

1) For gamma spectroscopy systems, background measurements shall be performed on at least a monthly basis.

2) For alpha spectroscopy systems, background measurements shall be performed on at least a monthly basis.

3) For gas-proportional counters background measurements shall be performed at least on a weekly basis.

4) For scintillation counters, background measurements shall be performed each day of use.

d) Instrument Contamination Monitoring

The laboratory shall have a written procedure for monitoring radiation measurement instrumentation for radioactive contamination. The procedure shall indicate the frequency of the monitoring and shall indicate criteria, which initiates corrective action.

D.4.5 Minimum Detectable Activity (MDA)/Minimum Detectable Concentration ~~Detection Limits~~

a) Must be determined prior to sample analysis and must be redetermined each time there is a significant change in the test method or instrument type.

b) The procedures employed must be documented and consistent with mandated method or regulation.

D.4.6 Data Reduction

a) Refer to Section 5.5.4.7.2, "Computers and Electronic Data Related Requirements," of this document.

b) Measurement Uncertainties - each result shall be reported with the associated measurement uncertainty. The procedures for determining the measurement uncertainty must be documented and be consistent with mandated method and regulation.

D.4.7 Quality of Standards and Reagents

a) The quality control program shall establish and maintain provisions for radionuclide standards.

1) Reference standards that are used in a radiochemical laboratory shall be obtained from the National Institute of Standards and Technology (NIST), ~~EPA,~~ or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.

2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22 - 1995, Section 8, Certificates.

3) Laboratories should consult with the supplier if the lab's verification of the activity of the reference traceable standard indicates a noticeable deviation from the certified value. The laboratory shall not use a value other than the decay corrected certified value. The laboratory shall have a written procedure for handling, storing and establishment of expiration dates for reference standards.

b) All reagents used shall be analytical reagent grade or better.

D.4.8 Constant and Consistent Test Conditions

The laboratory shall maintain a radiological control program that addresses analytical radiological control. The program shall address the procedures for segregating samples with potentially widely varying levels of radioactivity. The radiological control program shall explicitly define how low level and high level samples will be identified, segregated and processed in order to prevent sample cross-contamination.

~~a) To prevent incorrect analysis results caused by the spread of contamination among samples, the laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.~~

~~b) For gamma spectrometry systems, background check measurements shall be performed each day of use.~~

~~c) For alpha spectrometry systems, background check measurements shall be performed except when using the electro-plating method of sample preparation.~~

~~d) For gas-proportional counter systems, background check measurements shall be performed each day of use.~~

D.5 AIR TESTING

These standards shall apply to samples that are submitted to a laboratory for the purpose of analysis. They do not apply to field activities such as source air emission measurements or the use of continuous analysis devices.

D.5.1 Negative and Positive Controls

a) Negative Controls

1) Method Blanks – Shall be performed at a frequency of at least one (1) per batch of twenty (20) environmental samples or less per sample preparation method. The results of the method blank analysis shall be used to evaluate the contribution of the laboratory provided sampling media and analytical sample preparation procedures to the amount of analyte found in each sample. If the method blank result is greater than the limit of detection ~~detection limit~~ and contributes greater than 10% of the total amount of analyte found in the sample, the source of the contamination must be investigated and measures taken to eliminate the source of contamination. If contamination is found, the data shall be qualified in the report.

2) Collection Efficiency- Sampling trains consisting of multiple sections (e.g. filters, sorbent tubes, impingers) that are received intact by the laboratory, shall be separated into "front" and "back" sections if required by the client. Each section shall be processed and analyzed separately and the analytical results reported separately.

b) Positive Controls

1) Laboratory Control Sample (LCS) – Shall be analyzed at a rate of at least one (1) per batch of twenty (20) or fewer samples per sample preparation method for each analyte. If a spiking solution is not available, a calibration solution, whose concentration approximates that of the samples, shall be included in each batch and with each lot of media. If a calibration solution must be used for the LCS, the client will be notified prior to the start of analysis. The concentration of the LCS shall be relevant to the intended use of the data and either at a regulatory limit or below it.

c) Surrogates - Shall be used as required by the test method or if requested by the client.

d) Matrix spike – Shall be used as required by the test method, or if requested by the client.

D.5.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates – Shall be analyzed at a minimum of 1 in 20 samples per sample batch. The laboratory shall document their procedure to select the use of appropriate types of spikes and duplicates. The selected samples(s) shall be rotated among client samples so that various sample matrix problems may be noted and/or addressed. Poor performance in the spikes and duplicates may indicate a problem with the sample composition and shall be reported to the client.

D.5.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Demonstration of Capability – (Sections 5.5.2.6 and 5.5.4.2.2) shall be performed prior to the analysis of any samples and with a significant change in instrument type, personnel, quality system matrix, or test method.

b) Calibration – Calibration protocols specified in Section 5.5.5.2 shall be followed.

c) Proficiency Test Samples – The results of such analyses (5.4.1.5.k or 5.5.9.1)shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.5.4 Limit of Detection ~~Detection Limits~~

The laboratory shall utilize a test method that provides a limit of detection ~~detection limit~~ that is appropriate and relevant for the intended use of the data. Limit of detection ~~detection limits~~ shall be determined by the protocol in the mandated test method or applicable regulation, e.g., MDL. If the protocol for determining limit of detection ~~detection limits~~ is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

a) A limit of detection ~~detection limit~~ study is not required for any component for which spiking solutions are not available such as temperature or on-line analyses.

b) The limit of detection ~~detection limit~~ shall be initially determined for the compounds of interest in each test method in a quality system matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the limit of detection ~~detection limit~~ must be determined in the quality system matrix of interest (see definition of matrix).

c) Limit of detection ~~detection limits~~ must be determined each time there is a significant change in the test method or instrument type.

d) All sample processing steps of the analytical method must be included in the determination of the limit of detection ~~detection limit~~.

e) All procedures used must be documented. Documentation must include the quality system matrix type. All supporting data must be retained.

f) The laboratory must have established procedures to tie limit of detection ~~detection limits~~ with limit of quantitation ~~limits~~.

D.5.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.5.6 Quality of Standards and Reagents

a) The source of standards shall comply with 5.5.6.2.2.2.

b) The purity of each analyte standard and each reagent shall be documented by the laboratory through certificates of analyses from the manufacturer/vendor, manufacturer/vendor specifications, and/or independent analysis.

c) In methods where the purity of reagents is not specified, analytical reagent grade or higher quality, if available, shall be used.

D.5.7 Selectivity

The laboratory shall develop and document acceptance criteria for test method selectivity such as absolute and relative retention times, wavelength assignments, mass spectral library quality of match, and mass spectral tuning.

D.5.8 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) The laboratory shall document that all sampling equipment, containers and media used or supplied by the laboratory meet required test method criteria.

c) If supplied or used by the laboratory, procedures for field equipment decontamination shall be developed and their use documented.

d) The laboratory shall have a documented program for the calibration and verification of sampling equipment such as pumps, meter boxes, critical orifices,flow measurement devices and continuous analyzers, if these equipment are used or supplied by the laboratory.

D.6 ASBESTOS TESTING

These standards apply to laboratories undertaking the examination of asbestos samples. These standards are organized by analytical technique including transmission electron microscopy (TEM) for the analysis of water, wastewater, air, and bulk samples; phase contrast microscopy (PCM) for analysis of workplace air; and polarized light microscopy (PLM) for analysis of bulk samples. These procedures for asbestos analysis involve sample preparation followed by detection of asbestos. If NIST SRMs specified below are unavailable, the laboratory may substitute an equivalent reference material with a certificate of analysis.

D.6.1 Negative Controls

D.6.1.1 Transmission Electron Microscopy

D.6.1.1.1 Water and Wastewater

a) Blank determinations shall be made prior to sample collection. When using polyethylene bottles, one bottle from each batch, or a minimum of one from each 24 shall be tested for background level. When using glass bottles, four bottles from each 24 shall be tested. An acceptable bottle blank level is defined as ≤ 0.01 MFL > 10 μm. (EPA /600/R-94/134, Method 100.2, Section 8.2)

b) A process blank sample consisting of fiber-free water shall be run before the first field sample. The quantity of water shall be ≥ 10 mL for a 25-mm diameter filter and ≥ 50 mL for a 47-mm diameter filter. (EPA /600/R-94/134, Method 100.2, Section 11.8)

D.6.1.1.2 Air

a) A blank filter shall be prepared with each set of samples. A blank filter shall be left uncovered during preparation of the sample set and a wedge from that blank filter shall be prepared

alongside wedges from the sample filters. At minimum, the blank filter shall be analyzed for each 20 samples analyzed. (40 CFR Part 763, Appendix A to Subpart E (AHERA), Table 1)

b) Maximum contamination on a single blank filter shall be no more than 53 structures/mm^2. Maximum average contamination for all blank filters shall be no more than 18 structures/mm^2. (AHERA, III.F.2)

D.6.1.1.3 Bulk Samples

a) Contamination checks using asbestos-free material, such as the glass fiber blank in SRM 1866 (Page C-3, NIST Handbook 150-3, August 1994) shall be performed at a frequency of 1 for every 20 samples analyzed. The detection of asbestos at a concentration exceeding 0.1% will require an investigation to detect and remove the source of the asbestos contamination.

b) The laboratory must maintain a list of non-asbestos fibers that can be confused with asbestos (Section 7.5, Page C-8, NIST Handbook 150-3, August 1994). The list must include crystallographic and/or chemical properties that disqualify each fiber being identified as asbestos (Section 2.5.5.2.1 Identification, Page 54, EPA/600/R-93/116).

c) The laboratory should have a set of reference asbestos materials from which a set of reference diffraction and X-ray spectra have been developed.

D.6.1.2 Phase Contrast Microscopy

At least two (2) field blanks (or 10% of the total samples, whichever is greater) shall be submitted for analysis with each set of samples. Field blanks shall be handled in a manner representative of actual handling of associated samples in the set with a single exception that air shall not be drawn through the blank sample. A blank cassette shall be opened for approximately thirty (30) seconds at the same time other cassettes are opened just prior to analysis. Results from field blank samples shall be used in the calculation to determine final airborne fiber concentration. The identity of blank filters should be unknown to the counter until all counts have been completed. If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

D.6.1.3 Polarized Light Microscopy

a) Friable Materials - At least one blank slide must be prepared daily or with every 50 samples analyzed, whichever is less. This is prepared by mounting a subsample of an isotropic verified non-ACM (e.g., fiberglass in SRM 1866) in a drop of immersion oil (n_D should reflect usage of various n_D's) on a clean slide, rubbing preparation tools (forceps, dissecting needles, etc.) in the mount and placing a clean coverslip on the drop. The entire area under the coverslip must be scanned to detect any asbestos contamination. A similar check must be made after every 20 uses of each piece of homogenization equipment. An isotropic verified non-ACM must be homogenized in the clean equipment, a slide prepared with the material and the slide scanned for asbestos contamination. (This can be substituted for the blank slide mentioned in this section.)

b) Non-Friable Materials - At least one non-ACM non-friable material must be prepared and analyzed with every 20 samples analyzed. This non-ACM must go through the full preparation and analysis regimen for the type of analysis being performed.

D.6.2 Test Variability/Reproducibility

D.6.2.1 Transmission Electron Microscopy

Quality assurance analyses shall be performed regularly covering all time periods, instruments, tasks, and personnel. The selection of samples shall be random and samples of special interest may be included in the selection of samples for quality assurance analyses. When possible, the checks on personnel performance shall be executed without their prior knowledge. A disproportionate number of analyses shall not be performed prior to internal or external audits. It is recommended that a laboratory initially be at 100% quality control (all samples reanalyzed). The proportion of quality control samples can later be lowered gradually, as control indicates, to a minimum of 10%.

D.6.2.1.1 Water and Wastewater

All analyses must be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings. Quality assurance analyses shall not be postponed during periods of heavy workloads. The total number of QA samples and blanks must be greater than or equal to 10% of the total sample workload. Precision of analyses is related to concentration, as gleaned from interlaboratory proficiency testing. Relative standard deviations (RSD) for amphibole asbestos decreased from 50% at 0.8 MFL to 25% at 7 MFL in interlaboratory proficiency testing, while RSD for chrysotile was higher, 50% at 6 MFL.

a) Replicate – A second, independent analysis shall be performed on the same grids but on different grid openings than used in the original analysis of a sample. Results shall be within 1.5X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (EPA /600/R-94/134, Method 100.2, Table 2)

b) Duplicate – A second aliquot of sample shall be filtered through a second filter, prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (EPA /600/R-94/134, Method 100.2, Table 2)

c) Verified Analyses – A second, independent analysis shall be performed on the same grids and grid openings used in the original analysis of a sample. The two sets of results shall be compared according to Turner and Steel (NISTIR 5351). This shall be performed at a frequency of 1 per 20 samples. Qualified analysts must maintain an average of $\geq$ 80% true positives, $\leq$ 20% false negatives, and $\leq$ 10% false positives.

D.6.2.1.2 Air

All analyses must be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings.

The laboratory and TEM analysts must obtain mean analytical results on NIST SRM 1876b so that trimmed mean values fall within 80% of the lower limit and 110% of the upper limit of the 95% confidence limits as published on the certificate. These limits are derived from the allowable false positives and false negatives given in Section D.6.2.1.2c, Verified Analysis, below. SRM 1876b shall be analyzed a minimum of once per year by each TEM analyst.

The laboratory must have documentation demonstrating that TEM analysts correctly classify at least 90% of both bundles and single fibrils of asbestos structures greater than or equal to 1 μm in length in known standard materials traceable to NIST, such as NIST bulk asbestos SRM 1866.

Interlaboratory analyses shall be performed to detect laboratory bias. The frequency of interlaboratory verified analysis must correspond to a minimum of 1 per 200 grid square analyses for clients.

If more than 1 TEM is used for asbestos analysis, intermicroscope analyses must be performed to detect instrument bias.

a) Replicate – A second, independent analysis shall be performed in accordance with Section D.6.2.1.1.a. (AHERA, Table III)

b) Duplicate – A second wedge from a sample filter shall be prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (AHERA, Table III)

a) Verified Analyses – A second, independent analysis shall be performed on the same grids and grid openings in accordance with Section D.6.2.1.1.c. (AHERA, Table III)

D.6.2.1.3 Bulk Samples

Determination of precision and accuracy should follow guidelines in NISTIR 5951, Guide for Quality Control on the Qualitative and Quantitative Analysis of Bulk Asbestos Samples: Version 1. Because bulk samples with low (< 10%) asbestos content are the most problematic, a laboratory's quality control program should focus on such samples. At least 30% of a laboratory's QC analyses shall be performed on samples containing from 1% to 10% asbestos.

a) Intra-Analyst Precision - At least 1 out of 50 samples must be reanalyzed by the same analyst. For single analyst laboratories, at least 1 out of every 10 samples must be reanalyzed by the same analyst.

b) Inter-Analyst Precision - At least 1 out of 15 samples must be reanalyzed by another analyst. Inter-analyst results will require additional reanalysis, possibly including another analyst, to resolve discrepancies when classification (ACM vs. non-ACM) errors occur, when asbestos identification errors occur, or when inter-analyst precision is found to be unacceptable.

c) Inter-Laboratory Precision - The laboratory must participate in round robin testing with at least one other laboratory. Samples must be sent to this other lab at least four times per year. These samples must be samples previously analyzed as QC samples. Results of these analyses must be assessed in accordance with QC requirements. As a minimum, the QC requirements must address misclassifications (false positives, false negatives) and misidentification of asbestos types.

D.6.2.2 Phase Contrast Microscopy

a) Inter-Laboratory Precision – Each laboratory analyzing air samples for compliance determination shall implement an inter-laboratory quality assurance program that as a minimum includes participation of at least two (2) other independent laboratories. Each laboratory shall participate in round robin testing at least once every six (6) months with at least all the other laboratories in its inter-laboratory quality assurance group. Each laboratory shall submit slides typical of its own workload for use in this program. The round robin shall be designed and results analyzed using appropriate statistical methodology. Results of this QA program shall be posted in each laboratory to keep the microscopists informed.

b) Intra- and Inter-Analyst Precision – Each analyst shall select and count a prepared slide from a "reference slide library" on each day on which air counts are performed. Reference slides shall be prepared using well-behaved samples taken from the laboratory workload. Fiber densities shall cover the entire range routinely analyzed by the laboratory. These slides shall be counted by all analysts to establish an original standard deviation and corresponding limits of acceptability. Results from the daily reference sample analysis shall be compared to the statistically derived acceptance limits using a control chart or a database. It is recommended that the labels on the reference slides be periodically changed so that the analysts do not become familiar with the samples. Intra- and inter-analyst precision may be estimated from blind recounts on reference samples. Inter-analyst precision shall be posted in each laboratory to keep the microscopists informed.

D.6.2.3 Polarized Light Microscopy

Refer to Section D.6.2.1.3.

D.6.3 Other Quality Control Measures

D.6.3.1 Transmission Electron Microscopy

D.6.3.1.1 Water and Wastewater

a) Filter preparations shall be made from all six asbestos types from NIST SRMs 1866 and 1867. These preparations shall have concentrations between 1 and 20 structures (> 10μm) per 0.01 mm^2. One of these preparations shall be analyzed independently at a frequency of 1 per 100 samples analyzed. Results shall be evaluated as verified asbestos analysis in accordance with Turner and Steel (NISTIR 5351).

b) NIST SRM 1876b must be analyzed annually by each analyst. Results shall be evaluated in accordance with limits published for that SRM. Comment: This SRM is not strictly appropriate for waterborne asbestos but analysts can demonstrate general TEM asbestos competence by producing results within the published limits of this (the only recognized TEM counting standard) SRM.

D.6.3.1.2 Air

a) Filter preparations shall be made from all six asbestos types in accordance with Section D.6.3.1.1.a.

b) NIST SRM 1876b must be analyzed annually in accordance with Section D.6.3.1.1.b.

D.6.3.1.3 Bulk Samples

All analysts must be able to correctly identify the six regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite). Standards for the six asbestos types listed are available from NIST (SRMs 1866 and 1867). These materials can also be used as identification standards for AEM (Section 3.2.1 Qualitative Analysis, Page 57, EPA/600/R-93/116).

D.6.3.2 Phase Contrast Microscopy

a) Test for Non-Random Fiber Distribution - Blind recounts by the same analyst shall be performed on 10% of the filters counted. A person other than the counter should re-label slides before the second count. A test for type II error (NIOSH 7400, Issue 2, 15 August

1994, Section 13) shall be performed to determine whether a pair of counts by the same analyst on the same slide should be rejected due to non-random fiber distribution. If a pair of counts is rejected by this test, the remaining samples in the set shall be recounted and the new counts shall be tested against first counts. All rejected paired counts shall be discarded. It shall not be necessary to use this statistic on blank recounts.

b) All individuals performing airborne fiber analysis must have taken the NIOSH Fiber Counting Course for sampling and evaluating airborne asbestos dust or an equivalent course.

c) All laboratories shall participate in a national sample testing scheme such as the Proficiency Analytical Testing (PAT) program or the Asbestos Analysts Registry (AAR) program, both sponsored by the American Industrial Hygiene Association (AIHA), or equivalent.

D.6.3.3 Polarized Light Microscopy

a) Friable Materials - Because accuracy cannot be determined by reanalysis of routine field samples, at least 1 out of 100 samples must be a standard or reference sample that has been routinely resubmitted to determine analyst's precision and accuracy. A set of these samples should be accumulated from proficiency testing samples with predetermined weight compositions or from standards generated with weighed quantities of asbestos and other bulk materials (Perkins and Harvey, 1993; Parekh et al., 1992; Webber et al., 1982). At least half of the reference samples submitted for this QC must contain between 1 and 10% asbestos.

b) Non-Friable Materials - At least 1 out of 100 samples must be a verified quantitative standard that has routinely been resubmitted to determine analyst precision and accuracy.

D.6.4 Method Evaluation

In order to ensure the accuracy of reported results, the following procedures shall be in place:

a) Demonstration of Capability – (Refer to Section 5.10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, or method.

b) Performance Audits – (Refer to Section 5.4.2j or 5.5.3.4) The results of such analyses shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.6.5 Asbestos Measurement System Calibration

Refer to methods referenced in the following sections for specific equipment requirements.

D.6.5.1 Transmission Electron Microscopy

AEM (Analytical Electron Microscopy) equipment requirements will not be discussed in this document.

D.6.5.1.1 Water and Wastewater

All calibrations listed below (unless otherwise noted) must be performed under the same analytical conditions used for routine asbestos analysis and must be recorded in a notebook and include date and analyst's signature. Frequencies stated below may be reduced to "before next use" if no samples are analyzed after the last calibration period has expired. Likewise, frequencies may have to be increased following non-routine maintenance or unacceptable calibration performance.

a) Magnification Calibration – Magnification calibration must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 10,000 and 20,000x. A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification. Calibration data must be displayed on control charts that show trends over time. (EPA /600/R-94/134, Method 100.2, Section 10.1)

b) Camera Constant – The camera length of the TEM in the Selected Area Electron Diffraction (SAED) mode must be calibrated before SAED patterns of unknown samples are observed. The diffraction specimen must be at the eucentric position for this calibration. This calibration shall allow accurate (< 10% variation) measurement of layer-line spacings on the medium used for routine measurement, i.e., the phosphor screen or camera film. This must also allow accurate (< 5% variation) measurement of zone axis SAED patterns on permanent media, e.g., film. Calibrations shall be performed monthly to establish the stability of the camera constant (EPA /600/R-94/134, Method 100.2, Section 10.2). Where non-asbestiform minerals may be expected (e.g., winchite, richterite, industrial talc, vermiculite, etc.), an internal camera constant standard such as gold, shall be deposited and measured on each sample to facilitate accurate indexing of zone axis SAED patterns. In such cases, layer line analysis alone shall not be used. Calibration data must be displayed on control charts that show trends over time.

c) Spot Size – The diameter of the smallest beam spot at crossover must be less than 250 nm as calibrated quarterly. Calibration data must be displayed on control charts that show trends over time. (EPA /600/R-94/134, Method 100.2, Section 10.3)

d) Beam Dose - The beam dose shall be calibrated so that beam damage to chrysotile is minimized, specifically so that an electron diffraction pattern from a single fibril $\geq$1 μm in length from a NIST SRM chrysotile sample is stable in the electron beam dose for at least 15 seconds.

e) EDXA System

1) The x-ray energy vs. channel number for the EDXA system shall be calibrated to within 20 eV for at least two peaks between 0.7 keV and 10 keV. One peak shall be from the low end (0.7 keV to 2 keV) and the other peak from the high end (7 keV to 10 keV) of this range. The calibration of the x-ray energy shall be checked prior to each analysis of samples and recalibrated if out of the specified range.

2) The ability of the system to resolve the Na Kα line from the Cu L line shall be confirmed quarterly by obtaining a spectrum from the NIST SRM 1866 crocidolite sample on a copper grid.

3) The k-factors for elements found in asbestos (Na, Mg, Al, Si, Ca, and Fe) relative to Si shall be calibrated semiannually, or anytime the detector geometry may be altered. NIST SRM 2063a shall be used for Mg, Si, Ca, Fe, while k-factors for Na and Al may be obtained from suitable materials such as albite, kaersutite, or NIST SRM 99a. The k-factors shall be determined to a precision (2s) within 10% relative to the mean value obtained for Mg, Al, Si, Ca, and Fe, and within 20% relative to the mean value obtained for Na. The k-factor relative to Si for Na shall be between 1.0 and 4.0, for Mg and Fe shall be between 1.0 and 2.0, and for Al and Ca shall be between 1.0 and

1.75. The k-factor for Mg relative to Fe shall be 1.5 or less. Calibration data must be displayed on control charts that show trends over time.

4) The detector resolution shall be checked quarterly to ensure a full-width half-maximum resolution of < 175 eV at Mn Kα (5.90 keV). Calibration data must be displayed on control charts that show trends over time.

5) The portions of a grid in a specimen holder for which abnormal x-ray spectra are generated under routine asbestos analysis conditions shall be determined and these areas shall be avoided in asbestos analysis.

6) The sensitivity of the detector for collecting x-rays from small volumes shall be documented quarterly by collecting resolvable Mg and Si peaks from a unit fibril of NIST SRM 1866 chrysotile.

f) Low Temperature Asher - The low temperature asher shall be calibrated quarterly by determining a calibration curve for the weight vs. ashing time of collapsed mixed-cellulose-ester (MCE) filters. Calibration data must be displayed on control charts that show trends over time.

g) Grid Openings - The magnification of the grid opening measurement system shall be calibrated using an appropriate standard at a frequency of 20 openings/20 grids/lot of 1000 or 1 opening/sample. The variation in the calibration measurements (2s) is <5% of the mean calibration value.

D.6.5.1.2 Air

All calibrations must be performed in accordance with Section D.6.5.1.1, with the exception of magnification. Magnification calibration must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 15,000 to 20,000x (AHERA, III.G.1.c). A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification.

D.6.5.1.3 Bulk Samples

All calibrations must be performed in accordance with Section D.6.5.1.2.

D.6.5.2 Phase Contrast Microscopy

a) At least once daily, the analyst shall use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric.

b) The phase-shift limit of detection ~~detection limit~~ of the microscope shall be checked monthly or after modification or relocation using an HSE/NPL phase-contrast test slide for each analyst/microscope combination (refer to NIOSH 7400, Issue 2, 15 August 1994, Section 10b). This procedure assures that the minimum detectable fiber diameter (< ca. 0.25μm) for this microscope is achieved.

c) Prior to ordering the Walton-Beckett graticule, calibration, in accordance with NIOSH 7400, Issue 2, 15 August 1994, Appendix A, shall be performed to obtain a counting area 100 μm in diameter at the image plane. The diameter, d_c (mm), of the circular counting area and the

disc diameter must be specified when ordering the graticule. The field diameter (D) shall be verified (or checked), to a tolerance of 100 μm ± 2 μm, with a stage micrometer upon receipt of the graticule from the manufacturer. When changes (zoom adjustment, disassembly, replacement, etc.) occur in the eyepiece-objective-reticle combination, field diameter must be re-measured (or re-calibrated) to determine field area (mm^2). Re-calibration of field diameter shall also be required when there is a change in interpupillary distance (i.e., change in analyst). Acceptable range for field area shall be 0.00754 mm^2 to 0.00817 mm^2. The actual field area shall be documented and used.

D.6.5.3 Polarized Light Microscopy

a) Microscope Alignment - To accurately measure the required optical properties, a properly aligned polarized light microscope (PLM) shall be utilized. The PLM shall be aligned before each use. (Section 2.2.5.2.3, EPA/600/R-93/116, July 1993)

b) Refractive Index Liquids - Series of n_D = 1.49 through 1.72 in intervals less than or equal to 0.005. Refractive index liquids for dispersion staining, high- dispersion series 1.550, 1.605, 1.680. The accurate measurement of the refractive index (RI) of a substance requires the use of calibrated refractive index liquids. These liquids shall be calibrated at first use and semiannually, or next use, whichever is less frequent, to an accuracy of 0.004, with a temperature accuracy of 2°C using a refractometer or RI glass beads.

D.6.6 Analytical Sensitivity

D.6.6.1 Transmission Electron Microscopy

D.6.6.1.1 Water and Wastewater

An analytical sensitivity of 200,000 fibers per liter (0.2 MFL) is required for each sample analyzed (EPA /600/R-94/134, Method 100.2, Section 1.6). Analytical sensitivity is defined as the waterborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the fraction of the filter sampled and the dilution factor (if applicable).

D.6.6.1.2 Air

An analytical sensitivity of 0.005 structures/cm^2 is required for each sample analyzed. Analytical sensitivity is defined as the airborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the effective surface area of the filter, the filter area analyzed, and the volume of air sampled (AHERA, Table I).

D.6.6.1.3 Bulk Samples

a) The range is dependent on the type of bulk material being analyzed. The sensitivity may be as low as 0.0001% depending on the extent to which interfering materials can be removed during the preparation of AEM specimens. (Section 2.5.2 Range, Page 51, EPA/600/R-93/116)

b) There should be an error rate of less than 1% on the qualitative analysis for samples that contain chrysotile, amosite, and crocidolite. A slightly higher error rate may occur for samples that contain anthophyllite, actinolite, and tremolite, as it can be difficult to distinguish among the three types. (Section 3, Page 10, NIST Handbook 150-3, August 1994)

D.6.6.2 Phase Contrast Microscopy

The normal quantitative working range of the test method is 0.04 to 0.5 fiber/ cm^2 for a 1000 L air sample. An ideal counting range on the filter shall be 100 to 1300 fibers/mm^2. The limit of detection (LOD) is estimated to be 5.5 fibers per 100 fields or 7 fibers/mm^2. The LOD in fiber/cc will depend on sample volume and quantity of interfering dust but shall be <0.01 fiber/ cm^2 for atmospheres free of interferences. (NIOSH 7400, Issue 2, 15 August 1994)

D.6.6.3 Polarized Light Microscopy

The laboratory shall utilize a test method that provides a limit of detection ~~detection limit~~ that is appropriate and relevant for the intended use of the data. Limit of detection ~~detection limits~~ shall be determined by the protocol in the test method or applicable regulation.

D.6.7 Data Reduction

D.6.7.1 Transmission Electron Microscopy

D.6.7.1.1 Water and Wastewater

a) The concentration of asbestos in a given sample must be calculated in accordance with EPA /600/R-94/134, Method 100.2, Section 12.1. Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample (EPA /600/R-94/134, Method 100.2, Section 12.2.2).

D.6.7.1.2 Air

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized, e.g., AHERA. Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample.

D.6.7.1.3 Bulk Samples

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993). Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties - Proficiency testing for floor tiles analyzed by TEM following careful gravimetric reduction (New York ELAP Certification Manual Item 198.4) has revealed an interlaboratory standard deviation of approximately 20% for residues containing 70% or more asbestos. Standard deviations range from 20% to 60% for residues with lower asbestos content.

D.6.7.2 Phase Contrast Microscopy

a) Airborne fiber concentration in a given sample must be calculated in accordance with NIOSH 7400, Issue 2, 15 August 1994, Sections 20 and 21. Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the intra-laboratory and inter-laboratory relative standard deviation with each set of results. (NIOSH 7400, Issue 2, 15 August 1994)

c) Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with >50% of the filter area covered with particulate should be reported as "uncountable" or "probably biased". Other fiber counts outside the 100-1300 fibers/mm^2 range should be reported as having "greater than optimal variability" and as being "probably biased".

D.6.7.3 Polarized Light Microscopy

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993). Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Method Uncertainties - Precision and accuracy must be determined by the individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonable expanded errors (refer to EPA/600/R-93/116, July 1993, Table 2-1) should be generated for different concentrations of asbestos.

D.6.8 Quality of Standards and Reagents

D.6.8.1 Transmission Electron Microscopy

a) The quality control program shall establish and maintain provisions for asbestos standards.

   1) Reference standards that are used in an asbestos laboratory shall be obtained from the National Institute of Standards and Technology (NIST), EPA, or suppliers who participate in supplying NIST standards or NIST traceable asbestos. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.

   2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22-1995, Section 8, Certificates.

b) All reagents used shall be analytical reagent grade or better.

c) The laboratory shall have mineral fibers or data from mineral fibers that will allow differentiating asbestos from at least the following "look-alikes": fibrous talc, sepiolite, wollastonite, attapulgite (palygorskite), halloysite, vermiculite scrolls, antigorite, lizardite, pyroxenes, hornblende, richterite, winchite, or any other asbestiform minerals that are suspected as being present in the sample.

D.6.8.2 Phase Contrast Microscopy

Standards of known concentration have not been developed for this testing method. Routine workload samples that have been statistically validated and national proficiency testing samples such as PAT and AAR samples available from the AIHA may be utilized as reference samples (refer to Section D.6.2.2b) to standardize the optical system and analyst. All other testing reagents and devices (HSE/NPL test slide and Walton-Beckett Graticule) shall conform to the specifications of the method (refer to NIOSH 7400, Issue 2, 15 August 1994).

D.6.8.3 Polarized Light Microscopy

Refer to Section D.6.8.1.

D.6.9 Constant and Consistent Test Conditions

The laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.

QUALITY SYSTEMS
APPENDIX E

ADDITIONAL SOURCES OF INFORMATION AND ASSISTANCE

-Non-Mandatory Appendix-

Appendix E – ADDITIONAL SOURCES OF INFORMATION
Non-Mandatory Appendix

Additional sources of information are available to assist laboratories in the design and implementation of a quality system. These materials may be found on the NELAC web page at www.epa.gov/ttn/nelac under the topic “Related Information.”

~~QUALITY SYSTEMS~~
~~*APPENDIX F*~~

~~CROSS-REFERENCE TO NELAC 2001 QUALITY SYSTEMS CHAPTER 5~~

Appendix F - CROSS-REFERENCE TO NELAC 2001 QUALITY SYSTEMS CHAPTER 5

NELAC 2001 Chapter 5	NELAC 2001 text begins with:	NELAC 2002 Chapter 5 (ISO 17025 Format)
5	*QUALITY SYSTEMS*	5
5.1	*SCOPE*	5.1
5.1.a	*This Standard sets out the general requirements that a laboratory has*	5.1.1
5.1.b	*This Standard includes additional requirements and information for*	5.1.1 and 5.1.4
5.1.c	*This Standard is for use by environmental testing laboratories in the*	5.1.4
5.2	*REFERENCES*	5.2
5.3	*DEFINITIONS*	5.3
5.4	*ORGANIZATION AND MANAGEMENT*	5.4
5.4.1	*Legal Definition of Laboratory*	5.4.1.1 and 5.4.1.2
5.4.2	*Organization*	5.4.1.5
5.4.2.a	*have managerial staff with the authority and resources needed to*	5.4.1.5.a
5.4.2.b	*have processes to ensure that its personnel are free from any*	5.4.1.5.b
5.4.2.c	*be organized in such a way that confidence in its independence of*	5.4.1.5.d
5.4.2.d	*specify and document the responsibility, authority, and interrelationship*	5.4.1.5.f
5.4.2.d.1	*a clear description of the lines of responsibility in the laboratory and*	5.4.1.5.f
5.4.2.d.2	*job descriptions for all positions.*	5.5.2.4
5.4.2.e	*provide supervision by persons familiar with the calibration or test*	5.4.1.5.g
5.4.2.f	*have a technical director(s) (however named) who has overall*	5.4.1.5.h
5.4.2.g	*have a quality assurance officer (however named) who has*	5.4.1.5.i
5.4.2.g.1	*serve as the focal point for QA/QC and be responsible for the oversight*	5.4.1.5.i.1
5.4.2.g.2	*have functions independent from laboratory operations for which they*	5.4.1.5.i.2
5.4.2.g.3	*be able to evaluate data objectively and perform assessments without*	5.4.1.5.i.3
5.4.2.g.4	*have documented training and/or experience in QA/QC procedures and*	5.4.1.5.i.4
5.4.2.g.5	*have a general knowledge of the analytical test methods for which data*	5.4.1.5.i.5
5.4.2.g.6	*arrange for or conduct internal audits as per 5.5.3 annually; and,*	5.4.1.5.i.6
5.4.2.g.7	*notify laboratory management of deficiencies in the quality system and*	5.4.1.5.i.7
5.4.2.h	*nominate deputies in case of absence of the technical director(s) and/or*	5.4.1.5.j
5.4.2.i	*have documented policy and procedures to ensure the protection of*	5.4.1.5.c
5.4.2.j	*for purposes of qualifying for and maintaining accreditation, each*	5.4.1.5.k
5.5	*QUALITY SYSTEM - ESTABLISHMENT, AUDITS, ESSENTIAL*	5.4.2
5.5.1	*Establishment*	5.4.2.1
5.5.1.a	*The elements of this quality system shall be documented in the*	5.4.2.2
5.5.1.b	*The quality documentation shall be available for use by the laboratory*	5.4.2.1

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5.5.1.c	*The quality documentation shall be available for use by the laboratory*	5.4.2.2.a
5.5.1.d	*The laboratory management shall ensure that these policies and*	5.4.2.2.c and 5.4.2.2.d
5.5.1.e	*The quality manual shall be maintained current under the responsibility*	5.4.2.5
5.5.2	*Quality Manual*	5.4.2.3
5.5.2.a	*a quality policy statement, including objectives and commitments, by*	5.4.2.3.a
5.5.2.b	*the organization and management structure of the laboratory, its place*	5.4.2.3.b
5.5.2.c	*the relationship between management, technical operations, support*	5.4.2.3.c
5.5.2.d	*procedures to ensure that all records required under this Chapter are*	5.4.2.3.d
5.5.2.e	*job descriptions of key staff and reference to the job descriptions of*	5.4.2.3.e
5.5.2.f	*identification of the laboratory's approved signatories; at a minimum, the*	5.4.2.3.f
5.5.2.g	*the laboratory's procedures for achieving traceability of measurements;*	5.4.2.3.g
5.5.2.h	*a list of all test methods under which the laboratory performs its*	5.4.2.3.h
5.5.2.i	*mechanisms for ensuring that the laboratory reviews all new work to*	5.4.2.3.i
5.5.2.j	*reference to the calibration and/or verification test procedures used;*	5.4.2.3.j
5.5.2.k	*procedures for handling submitted samples;*	5.4.2.3.k
5.5.2.l	*reference to the major equipment and reference measurement*	5.4.2.3.l
5.5.2.m	*reference to procedures for calibration, verification and maintenance of*	5.4.2.3.m
5.5.2.n	*reference to verification practices which may include interlaboratory*	5.4.2.3.n
5.5.2.o	*procedures to be followed for feedback and corrective action whenever*	5.4.2.3.o
5.5.2.p	*the laboratory management arrangements for exceptionally permitting*	5.4.2.3.p
5.5.2.q	*procedures for dealing with complaints;*	5.4.2.3.q
5.5.2.r	*procedures for protecting confidentiality (including national security*	5.4.2.3.r
5.5.2.s	*procedures for audits and data review;*	5.4.2.3.s
5.5.2.t	*processes/procedures for establishing that personnel are adequately*	5.4.2.3.t
5.5.2.u	*ethics policy statement developed by the laboratory and*	5.4.2.3.u
5.5.2.v	*reference to procedures for reporting analytical results; and,*	5.4.2.3.v
5.5.2.w	*a Table of Contents, and applicable lists of references and glossaries,*	5.4.2.3.w
5.5.3	*Audits, Reviews and Corrective Actions*	5.4.13 and 5.4.14
5.5.3.1	*Internal Audits*	5.4.13.1 and 5.4.13.2
5.5.3.2	*Managerial Review*	5.4.14.1 and 5.4.14.2
5.5.3.3	*Audit Review*	5.4.13.3
5.5.3.4	*Performance Audits*	5.5.9.1
5.5.3.4.a	*internal quality control procedures using statistical techniques; (see*	5.5.9.1
5.5.3.4.b	*participation in proficiency testing or other interlaboratory comparisons*	5.5.9.1.b
5.5.3.4.c	*use of certified reference materials and/or in-house quality control using*	5.5.9.1.a

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5.5.3.4.d	*replicate testings using the same or different test methods;*	5.5.9.1.c
5.5.3.4.e	*re-testing of retained samples;*	5.5.9.1.d
5.5.3.4.f	*correlation of results for different but related analysis of a sample (for*	5.5.9.1.e
5.5.3.5	*Corrective Actions*	5.4.10.6
5.5.3.5.a	*In addition to providing acceptance criteria and specific protocols for*	5.4.10.6.a
5.5.3.5.a.1	*identify the individual(s) responsible for assessing each QC data type;*	5.4.10.6.a.1
5.5.3.5.a.2	*identify the individual(s) responsible for initiating and/or recommending*	5.4.10.6.a.2
5.5.3.5.a.3	*identify the individual(s) responsible for initiating and/or recommending*	5.4.10.6.a.3
5.5.3.5.a.4	*specify how out-of-control situations and subsequent corrective actions*	5.4.10.6.a.4
5.5.3.5.a.5	*specify procedures for management (including the QA officer) to review*	5.4.10.6.a.5
5.5.3.5.b	*To the extent possible, samples shall be reported only if all quality*	5.4.10.6.b
5.5.4	*Essential Quality Control Procedures*	5.5.9.2
5.5.4.a	*All laboratories shall have detailed written protocols in place to monitor*	5.5.9.2.a
5.5.4.a.1	*Positive and negative controls to monitor tests such as blanks, spikes,*	5.5.9.2.a.1
5.5.4.a.2	*Tests to define the variability and/or repeatability of the laboratory*	5.5.9.2.a.2
5.5.4.a.3	*Measures to assure the accuracy of the test method including*	5.5.9.2.a.3
5.5.4.a.4	*Measures to evaluate test method capability, such as detection limits*	5.5.9.2.a.4
5.5.4.a.5	*Selection of appropriate formulae to reduce raw data to final results*	5.5.9.2.a.5
5.5.4.a.6	*Selection and use of reagents and standards of appropriate quality;*	5.5.9.2.a.6
5.5.4.a.7	*Measures to assure the selectivity of the test for its intended purpose;*	5.5.9.2.a.7
5.5.4.a.8	*Measures to assure constant and consistent test conditions (both*	5.5.9.2.a.8
5.5.4.b	*All quality control measures shall be assessed and evaluated on an on-*	5.5.9.2.b
5.5.4.c	*The laboratory shall have procedures for the development of*	5.5.9.2.c
5.5.4.d	*The quality control protocols specified by the laboratory's method*	5.5.9.2.d
5.6	*PERSONNEL*	5.5.2
5.6.1	*General Requirements for Laboratory Staff*	5.5.2.1
5.6.2	*Laboratory Management Responsibilities*	5.5.2.6
5.6.2.a	*Defining the minimal level of qualification, experience and skills*	5.5.2.6.a
5.6.2.b	*Ensuring that all technical laboratory staff have demonstrated capability*	5.5.2.6.b
5.6.2.c	*Ensuring that the training of each member of the technical staff is kept*	5.5.2.6.c
5.6.2.c.1	*Evidence must be on file that demonstrates that each employee has*	5.5.2.6.c.1
5.6.2.c.2	*Training courses or workshops on specific equipment, analytical*	5.5.2.6.c.2
5.6.2.c.3	*Training courses in ethical and legal responsibilities including the*	5.5.2.6.c.3
5.6.2.c.4	*Analyst training shall be considered up to date if an employee training*	5.5.2.6.c.4
5.6.2.c.4.i	*Acceptable performance of a blind sample (single blind to the analyst);*	5.5.2.6.c.4.i

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5.6.2.c.4.ii	*Another demonstration of capability;*	5.5.2.6.c.4.ii
5.6.2.c.4.iii	*Successful analysis of a blind performance sample on a similar test*	5.5.2.6.c.4.iii
5.6.2.c.4.iv	*At least four consecutive laboratory control samples with acceptable*	5.5.2.6.c.4.iv
5.6.2.c.4.v	*If i-iv cannot be performed, analysis of authentic samples with results*	5.5.2.6.c.4.v
5.6.2.d	*Documenting all analytical and operational activities of the laboratory;*	5.5.2.6.d
5.6.2.e	*Supervising all personnel employed by the laboratory;*	5.5.2.6.e
5.6.2.f	*Ensuring that all sample acceptance criteria (Section 5.11) are verified*	5.5.2.6.f
5.6.2.g	*Documenting the quality of all data reported by the laboratory; and*	5.5.2.6.g
5.6.2.h	*Developing a proactive program for prevention and detection of*	5.5.2.6.h
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5.7	*PHYSICAL FACILITIES - ACCOMMODATION AND ENVIRONMENT*	5.5.3
5.7.1	*Environment*	5.5.3.1
5.7.1.a	*Laboratory accommodation, test areas, energy sources, lighting,*	5.5.3.1
5.7.1.b	*The environment in which these activities are undertaken shall not*	5.5.3.1
5.7.1.c	*The laboratory shall provide for the effective monitoring, control and*	5.5.3.2
5.7.1.d	*In instances where monitoring or control of any of the above mentioned*	5.1.5 and 5.5.3.2
5.7.2	*Work Areas*	5.5.3.3
5.7.2.a	*There shall be effective separation between neighboring areas when*	5.5.3.3
5.7.2.b	*Access to and use of all areas affecting the quality of these activities*	5.5.3.4
5.7.2.c	*Adequate measures shall be taken to ensure good housekeeping in the*	5.5.3.5
5.7.2.d	*Work spaces must be available to ensure an unencumbered work area.*	5.5.3.5
5.7.2.d.1	*access and entryways to the laboratory;*	5.5.3.5.a
5.7.2.d.2	*sample receipt area(s);*	5.5.3.5.b
5.7.2.d.3	*sample storage area(s);*	5.5.3.5.c
5.7.2.d.4	*chemical and waste storage area(s); and,*	5.5.3.5.d
5.7.2.d.5	*data handling and storage area(s).*	5.5.3.5.e
5.8	*EQUIPMENT AND REFERENCE MATERIALS*	5.5.5
5.8.a	*The laboratory shall be furnished with all items of equipment (including*	5.5.5.1
5.8.b	*All equipment shall be properly maintained, inspected and cleaned.*	5.5.5.3
5.8.c	*Any item of the equipment which has been subjected to overloading or*	5.5.5.7
5.8.d	*Each item of equipment including reference materials shall be labeled,*	5.5.6.4.c, 5.5.6.4.d, and 5.5.5.8
5.8.e	*Records shall be maintained of each major item of equipment and all*	5.5.5.5, 5.5.5.5.g, and 5.5.6.4.a
5.8.e.1	*the name of the item of equipment;*	5.5.5.5.a
5.8.e.2	*the manufacturer's name, type identification, and serial number or other*	5.5.5.5.b
5.8.e.3	*date received and date placed in service (if available);*	5.5.5.5.i

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5.8.e.4	*current location, where appropriate;*	5.5.5.5.d
5.8.e.5	*if available, condition when received (e.g. new, used, reconditioned);*	5.5.5.5.j
5.8.e.6	*copy of the manufacturer's instructions, where available;*	5.5.5.5.e
5.8.e.7	*dates and results of calibrations and/or verifications and date of the*	5.5.5.5.f
5.8.e.8	*details of maintenance carried out to date and planned for the future;*	5.5.5.5.g
5.8.e.9	*history of any damage, malfunction, modification or repair.*	5.5.5.5.h
5.9	*MEASUREMENT TRACEABILITY AND CALIBRATION*	5.5.6
5.9.1	*General Requirements*	5.5.6.1
5.9.2	*Traceability of Calibration*	5.5.6.2.2.2
5.9.2.a	*The overall program of calibration and/or verification and validation of*	5.5.6.2.2.2.a
5.9.2.b	*Calibration certificates shall indicate the traceability to national*	5.5.6.2.2.2.b
5.9.2.c	*Where traceability to national standards of measurement is not*	5.5.6.2.2.2.c
5.9.3	*Reference Standards*	5.5.6.3
5.9.3.a	*Reference standards of measurement held by the laboratory (such as*	5.5.6.3.1
5.9.3.b	*There shall be a program of calibration and verification for reference*	5.5.6.3.1
5.9.3.c	*Where relevant, reference standards and measuring and testing*	5.5.6.3.2
5.9.4	*Calibration*	5.5.5.2
5.9.4.1	*Support Equipment*	5.5.5.2.1
5.9.4.1.a	*All support equipment shall be maintained in proper working order. The*	5.5.5.2.1.a
5.9.4.1.b	*All support equipment shall be calibrated or verified at least annually,*	5.5.5.2.1.b
5.9.4.1.b.1	*The equipment shall be removed from service until repaired; or*	5.5.5.2.1.b.1
5.9.4.1.b.2	*The laboratory shall maintain records of established correction factors*	5.5.5.2.1.b.2
5.9.4.1.c	*Raw data records shall be retained to document equipment*	5.5.5.2.1.c
5.9.4.1.d	*Prior to use on each working day, balances, ovens, refrigerators,*	5.5.5.2.1.d
5.9.4.1.e	*Mechanical volumetric dispensing devices including burettes (except*	5.5.5.2.1.e
5.9.4.1.f	*For chemical tests the temperature, cycle time, and pressure of each*	5.5.5.2.1.f
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5.9.4.2.1	*Initial Instrument Calibration*	5.5.5.2.2.1
5.9.4.2.1.a	*The details of the initial instrument calibration procedures including*	5.5.5.2.2.1.a
5.9.4.2.1.b	*Sufficient raw data records must be retained to permit reconstruction of*	5.5.5.2.2.1.b
5.9.4.2.1.c	*Sample results must be quantitated from the initial instrument*	5.5.5.2.2.1.c
5.9.4.2.1.d	*All initial instrument calibrations must be verified with a standard*	5.5.5.2.2.1.d
5.9.4.2.1.e	*Criteria for the acceptance of an initial instrument calibration must be*	5.5.5.2.2.1.e
5.9.4.2.1.f	*Results of samples not bracketed by initial instrument calibration*	5.5.5.2.2.1.f

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5.9.4.2.1.g	*If the initial instrument calibration results are outside established*	5.5.5.2.2.1.g
5.9.4.2.1.h	*Calibration standards must include concentrations at or below the*	5.5.5.2.2.1.h
5.9.4.2.1.i	*If a reference or mandated method does not specify the number of*	5.5.5.2.2.1.i
5.9.4.2.2	*Continuing Instrument Calibration Verification*	5.5.5.10
5.9.4.2.2.a	*The details of the continuing instrument calibration procedure,*	5.5.5.10.a
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5.9.4.2.2.c	*Sufficient raw data records must be retained to permit reconstruction of*	5.5.5.10.c
5.9.4.2.2.d	*Criteria for the acceptance of a continuing instrument calibration*	5.5.5.10.d
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5.9.4.2.2.e.i	*When the acceptance criteria for the continuing calibration verification*	5.5.5.10.e.i
5.9.4.2.2.e.ii	*When the acceptance criteria for the continuing calibration verification*	5.5.5.10.e.ii
5.10	*TEST METHODS AND STANDARD OPERATING PROCEDURES*	5.5.4
5.10.1	*Methods Documentation*	5.5.4.1
5.10.1.a	*The laboratory shall have documented instructions on the use and*	5.5.4.1
5.10.1.b	*All instructions, standards, manuals and reference data relevant to the*	5.5.4.1
5.10.1.1	*Standard Operating Procedures (SOPs)*	5.5.4.1.1
5.10.1.1.a	*These documents, for example, may be equipment manuals provided*	5.5.4.1.1.a
5.10.1.1.b	*The test methods may be copies of published methods as long as any*	5.5.4.1.1.b
5.10.1.1.c	*Copies of all SOPs shall be accessible to all personnel.*	5.5.4.1.1.c
5.10.1.1.d	*The SOPs shall be organized .*	5.5.4.1.1.d
5.10.1.1.e	*Each SOP shall clearly indicate the effective date of the document, the*	5.5.4.1.1.e
5.10.1.2	*Laboratory Method Manual(s)*	5.5.4.1.2
5.10.1.2.a	*The laboratory shall have and maintain an in-house methods manual(s)*	5.5.4.1.2.a
5.10.1.2.b	*This manual may consist of copies of published or referenced test*	5.5.4.1.2.b
5.10.1.2.b.1	*identification of the test method;*	5.5.4.1.2.b.1
5.10.1.2.b.2	*applicable matrix or matrices;*	5.5.4.1.2.b.2
5.10.1.2.b.3	*detection limit;*	5.5.4.1.2.b.3
5.10.1.2.b.4	*scope and application, including components to be analyzed;*	5.5.4.1.2.b.4
5.10.1.2.b.5	*summary of the test method;*	5.5.4.1.2.b.5
5.10.1.2.b.6	*definitions;*	5.5.4.1.2.b.6
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5.10.1.2.b.8	*safety;*	5.5.4.1.2.b.8
5.10.1.2.b.9	*equipment and supplies;*	5.5.4.1.2.b.9
5.10.1.2.b.10	*reagents and standards;*	5.5.4.1.2.b.10
5.10.1.2.b.11	*sample collection, preservation, shipment and storage;*	5.5.4.1.2.b.11

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5.10.1.2.b.13	*calibration and standardization;*	5.5.4.1.2.b.13
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5.10.1.2.b.15	*calculations;*	5.5.4.1.2.b.15
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5.10.1.2.b.18	*data assessment and acceptance criteria for quality control measures;*	5.5.4.1.2.b.18
5.10.1.2.b.19	*corrective actions for out-of-control data;*	5.5.4.1.2.b.19
5.10.1.2.b.20	*contingencies for handling out-of-control or unacceptable data;*	5.5.4.1.2.b.20
5.10.1.2.b.21	*waste management;*	5.5.4.1.2.b.21
5.10.1.2.b.22	*references; and,*	5.5.4.1.2.b.22
5.10.1.2.b.23	*any tables, diagrams, flowcharts and validation data.*	5.5.4.1.2.b.23
5.10.2	*Test Methods*	5.5.4.2.1.c
5.10.2.a	*When the use of reference test methods for a sample analysis are*	5.5.4.2.1.b
5.10.2.b	*Where test methods are employed that are not required, as in the*	5.5.4.2.1.c
5.10.2.1	*Demonstration of Capability*	5.5.4.2.2
5.10.2.1.a	*Prior to acceptance and institution of any test method, satisfactory*	5.5.4.2.2.a
5.10.2.1.b	*Thereafter, continuing demonstration of method performance, as per*	5.5.4.2.2.b
5.10.2.1.c	*In cases where a laboratory analyzes samples using a test method that*	5.5.4.2.2.c
5.10.2.1.d	*In all cases, the appropriate forms such as the Certification Statement*	5.5.4.2.2.d
5.10.2.1.e	*A demonstration of capability must be completed each time there is a*	5.5.4.2.2.e
5.10.2.1.f	*In laboratories with a specialized "work cell(s)" (a group consisting of*	5.5.4.2.2.f
5.10.2.1.g	*When a work cell(s) is employed, and the members of the cell change,*	5.5.4.2.2.g
5.10.2.1.h	*When a work cell(s) is employed the performance of the group must be*	5.5.4.2.2.h
5.10.3	*Sample Aliquots*	5.5.7.1
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5.10.4.a	*The laboratory shall establish Standard Operating Procedure to ensure*	5.5.4.7.1.a
5.10.4.b	*The laboratory shall establish Standard Operating Procedures to*	5.5.4.7.1.b
5.10.4.c	*The laboratory shall establish Standard Operating Procedures*	5.5.4.7.1.c
5.10.5	*Documentation and Labeling of Standards and Reagents*	5.5.6.4
5.10.5.a	*The laboratory shall retain records for all standards, reagents and*	5.5.6.4.a
5.10.5.b	*Original containers (such as provided by the manufacturer or vendor)*	5.5.6.4.b
5.10.5.c	*Records shall be maintained on reagent and standard preparation.*	5.5.6.4.c
5.10.5.d	*All containers of prepared reagents and standards must bear a unique*	5.5.6.4.d
5.10.6	*Computers and Electronic Data Related Requirements*	5.5.4.7.2

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5.10.6.a	*all requirements of this Standard (i.e. Chapter 5) are met;*	5.5.4.7.2
5.10.6.b	*computer software is tested and documented to be adequate for use,*	5.5.4.7.2.a
5.10.6.c	*procedures are established and implemented for protecting the integrity*	5.5.4.7.2.b
5.10.6.d	*computer and automated equipment are maintained to ensure proper*	5.5.4.7.2.c
5.10.6.e	*it establishes and implements appropriate procedures for the*	5.5.4.7.2.d
5.11	*SAMPLE HANDLING, SAMPLE ACCEPTANCE POLICY AND*	5.5.8
5.11.1	*Sample Tracking*	5.5.8.2
5.11.1.a	*The laboratory shall have a documented system for uniquely identifying*	5.5.8.2.a
5.11.1.b	*This laboratory code shall maintain an unequivocal link with the unique*	5.5.8.2.b
5.11.1.c	*The laboratory ID code shall be placed on the sample container as a*	5.5.8.2.c
5.11.1.d	*The laboratory ID code shall be entered into the laboratory records (see*	5.5.8.2.d
5.11.1.e	*In cases where the sample collector and analyst are the same*	5.5.8.2.e
5.11.2	*Sample Acceptance Policy*	5.5.8.3.2
5.11.2.a	*Proper, full, and complete documentation, which shall include sample*	5.5.8.3.2.a
5.11.2.b	*Proper sample labeling to include unique identification and a labeling*	5.5.8.3.2.b
5.11.2.c	*Use of appropriate sample containers;*	5.5.8.3.2.c
5.11.2.d	*Adherence to specified holding times;*	5.5.8.3.2.d
5.11.2.e	*Adequate sample volume. Sufficient sample volume must be available*	5.5.8.3.2.e
5.11.2.f	*Procedures to be used when samples show signs of damage,*	5.5.8.3.2.f
5.11.3	*Sample Receipt Protocols*	5.5.8.3.1
5.11.3.a	*Upon receipt, the condition of the sample, including any abnormalities*	5.5.8.3.1.a and 5.5.8.3
5.11.3.a.1	*All samples which require thermal preservation shall be considered*	5.5.8.3.1.a.1
5.11.3.a.2	*The laboratory shall implement procedures for checking chemical*	5.5.8.3.1.a.2
5.11.3.b	*The results of all checks shall be recorded.*	5.5.8.3.1.b
5.11.3.c	*Where there is any doubt as to the item's suitability for testing, where*	5.5.8.3.1.c
5.11.3.c.1	*Retain correspondence and/or records of conversations concerning the*	5.5.8.3.1.c.1
5.11.3.c.2	*Fully document any decision to proceed with the analysis of samples*	5.5.8.3.1.c.2
5.11.3.c.2.i	*The condition of these samples shall, at a minimum, be noted on the*	5.5.8.3.1.c.2.i
5.11.3.c.2.ii	*The analysis data shall be appropriately "qualified" on the final report.*	5.5.8.3.1.c.2.ii
5.11.3.d	*The laboratory shall utilize a permanent chronological record such as a*	5.5.8.3.1.d
5.11.3.d.1	*This sample receipt log shall record the following:*	5.5.8.3.1.d.1
5.11.3.d.1.i	*Client/Project Name,*	5.5.8.3.1.d.1.i
5.11.3.d.1.ii	*Date and time of laboratory receipt,*	5.5.8.3.1.d.1.ii
5.11.3.d.1.iii	*Unique laboratory ID code (see 5.11.1), and,*	5.5.8.3.1.d.1.iii
5.11.3.d.1.iv	*Signature or initials of the person making the entries.*	5.5.8.3.1.d.1.iv

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5.11.3.d.2	*During the log-in process, the following information must be*	5.5.8.3.1.d.2
5.11.3.d.2.i	*The field ID code which identifies each container must be linked to the*	5.5.8.3.1.d.2.i
5.11.3.d.2.ii	*The date and time of sample collection must be linked to the sample*	5.5.8.3.1.d.2.ii
5.11.3.d.2.iii	*The requested analyses (including applicable approved test method*	5.5.8.3.1.d.2.iii
5.11.3.d.2.iv	*Any comments resulting from inspection for sample rejection shall be*	5.5.8.3.1.d.2.iv
5.11.3.e	*All documentation, such as memos or transmittal forms, that is*	5.5.8.3.1.e
5.11.3.f	*A complete chain of custody record form (Sections 5.12.3 and Appendix*	5.5.8.3.1.f
5.11.4	*Storage Conditions*	5.5.8.4
5.11.4.a	*Samples shall be stored according to the conditions specified by*	5.5.8.4.a
5.11.4.a.1	*Samples which require thermal preservation shall be stored under*	5.5.8.4.a.1
5.11.4.a.2	*Samples shall be stored away from all standards, reagents, food and*	5.5.8.4.a.2
5.11.4.b	*Sample fractions, extracts, leachates and other sample preparation*	5.5.8.4.b
5.11.4.c	*Where a sample or portion of the sample is to be held secure (for*	5.5.8.4.b
5.11.5	*Sample Disposal*	5.5.8.4.c
5.12	*RECORDS*	5.4.12
5.12.1	*Record Keeping System and Design*	5.4.12.1.5
5.12.1.a	*The records shall include the identity of personnel involved in sampling,*	5.4.12.1.5.a
5.12.1.b	*All information relating to the laboratory facilities equipment, analytical*	5.4.12.1.5.b
5.12.1.c	*The record keeping system shall facilitate the retrieval of all working*	5.4.12.1.5.c
5.12.1.d	*All changes to records shall be signed or initialed by responsible staff.*	5.4.12.1.5.d
5.12.1.e	*All generated data except those that are generated by automated data*	5.4.12.1.5.e
5.12.1.f	*Entries in records shall not be obliterated by methods such as erasures,*	5.4.12.1.5.f
5.12.1.g	*Refer to 5.10.6 for Computer and Electronic Data.*	5.4.12.1.5.g
5.12.2	*Records Management and Storage*	5.4.12.2.4
5.12.2.a	*All records (including those pertaining to calibration and test*	5.4.12.2.4.a
5.12.2.b	*All records, including those specified in 5.12.3 shall be retained for a*	5.4.12.2.4.b
5.12.2.c	*Records that are stored or generated by computers or personal*	5.4.12.2.4.c
5.12.2.d	*The laboratory shall establish a record management system for control*	5.4.12.2.4.d
5.12.2.e	*Access to archived information shall be documented with an access*	5.4.12.2.4.e
5.12.2.f	*The laboratory shall have a plan to ensure that the records are*	5.4.12.2.4.f
5.12.3	*Laboratory Sample Tracking*	5.4.12.2.5
5.12.3.1	*Sample Handling*	5.4.12.2.5.1
5.12.3.1.a	*Sample preservation including appropriateness of sample container and*	5.4.12.2.5.1.a
5.12.3.1.b	*Sample identification, receipt, acceptance or rejection and log-in;*	5.4.12.2.5.1.b
5.12.3.1.c	*Sample storage and tracking including shipping receipts, sample*	5.4.12.2.5.1.c

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5.12.3.1.d	*The laboratory shall have documented procedures for the receipt and*	5.4.12.2.5.1.d
5.12.3.2	*Laboratory Support Activities*	5.4.12.2.5.2
5.12.3.2.a	*All original raw data, whether hard copy or electronic, for calibrations,*	5.4.12.2.5.2.a
5.12.3.2.b	*A written description or reference to the specific test method used*	5.4.12.2.5.2.b
5.12.3.2.c	*Copies of final reports;*	5.4.12.2.5.2.c
5.12.3.2.d	*Archived standard operating procedures;*	5.4.12.2.5.2.d
5.12.3.2.e	*Correspondence relating to laboratory activities for a specific project;*	5.4.12.2.5.2.e
5.12.3.2.f	*All corrective action reports, audits and audit responses;*	5.4.12.2.5.2.f
5.12.3.2.g	*Proficiency test results and raw data; and,*	5.4.12.2.5.2.g
5.12.3.2.h	*Results of data review, verification, and cross-checking procedures.*	5.4.12.2.5.2.h
5.12.3.3	*Analytical Records*	5.4.12.2.5.3
5.12.3.3.a	*Laboratory sample ID code;*	5.4.12.2.5.3.a
5.12.3.3.b	*Date of analysis and time of analysis is required if the holding time is 72*	5.4.12.2.5.3.b
5.12.3.3.c	*Instrumentation identification and instrument operating*	5.4.12.2.5.3.c
5.12.3.3.d	*Analysis type;*	5.4.12.2.5.3.d
5.12.3.3.e	*All manual calculations, e.g., manual integrations; and,*	5.4.12.2.5.3.e
5.12.3.3.f	*Analyst's or operator's initials/signature;*	5.4.12.2.5.3.f
5.12.3.3.g	*Sample preparation including cleanup, separation protocols, incubation*	5.4.12.2.5.3.g
5.12.3.3.h	*Sample analysis;*	5.4.12.2.5.3.h
5.12.3.3.i	*Standard and reagent origin, receipt, preparation, and use;*	5.4.12.2.5.3.i
5.12.3.3.j	*Calibration criteria, frequency and acceptance criteria;*	5.4.12.2.5.3.j
5.12.3.3.k	*Data and statistical calculations, review, confirmation, interpretation,*	5.4.12.2.5.3.k
5.12.3.3.l	*Quality control protocols and assessment;*	5.4.12.2.5.3.l
5.12.3.3.m	*Electronic data security, software documentation and verification,*	5.4.12.2.5.3.m
5.12.3.3.n	*Method performance criteria including expected quality control*	5.4.12.2.5.3.n
5.12.3.4	*Administrative Records*	5.4.12.2.5.4
5.12.3.4.a	*Personnel qualifications, experience and training records;*	5.4.12.2.5.4.a
5.12.3.4.b	*Records of demonstration of capability for each analyst; and*	5.4.12.2.5.4.b
5.12.3.4.c	*A log of names, initials and signatures for all individuals who are*	5.4.12.2.5.4.c
5.13	*LABORATORY REPORT FORMAT AND CONTENTS*	5.5.10.1
5.13.a	*Except as discussed in 5.13.b, each report to an outside client shall*	5.5.10.2
5.13.a.1	*a title, e.g., "Test Report", or "Test Certificate", "Certificate of Results"*	5.5.10.2.a
5.13.a.2	*name and address of laboratory, and location where the test was*	5.5.10.2.b
5.13.a.3	*unique identification of the certificate or report (such as serial number)*	5.5.10.2.c
5.13.a.3.i	*The total number of pages may be listed on the first page of the report*	5.5.10.2.c.i

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5.13.a.3.ii	*Each page is identified with the unique report identification, the pages*	5.5.10.2.c.ii
5.13.a.4	*name and address of client, where appropriate and project name if*	5.5.10.2.d
5.13.a.5	*description and unambiguous identification of the tested sample*	5.5.10.2.f
5.13.a.6	*identification of test results derived from any sample that did not meet*	5.5.10.3.1.b
5.13.a.7	*date of receipt of sample, date and time of sample collection, date(s) of*	5.5.10.2.g
5.13.a.8	*identification of the test method used, or unambiguous description of*	5.5.10.2.e
5.13.a.9	*if the laboratory collected the sample, reference to sampling procedure;*	5.5.10.2.h
5.13.a.10	*any deviations from (such as failed quality control), additions to or*	5.5.10.3.1.a
5.13.a.11	*measurements, examinations and derived results, supported by tables,*	5.5.10.2.i
5.13.a.12	*when required, a statement of the estimated uncertainty of the test*	5.5.10.3.1.c
5.13.a.13	*a signature and title, or an equivalent electronic identification of the*	5.5.10.2.j
5.13.a.14	*at the laboratory's discretion, a statement to the effect that the results*	5.5.10.2.k
5.13.a.15	*at the laboratory's discretion, a statement that the certificate or report*	5.5.10.2.l
5.13.a.16	*clear identification of all test data provided by outside sources, such as*	5.5.10.6
5.13.a.17	*clear identification of numerical results with values outside of*	5.5.10.3.1.f
5.13.b	*Laboratories that are operated by a facility and whose sole function is to*	5.5.10.1
5.13.b.1	*The in-house laboratory is itself responsible for preparing the regulatory*	5.5.10.1.a
5.13.b.2	*The laboratory provides information to another individual within the*	5.5.10.1.b
5.13.c	*Where the certificate or report contains results of tests performed by*	5.5.10.6
5.13.d	*After issuance of the report, the laboratory report shall remain*	5.5.10.9
5.13.e	*The laboratory shall notify clients promptly, in writing, of any event such*	5.4.13.2
5.13.f	*The laboratory shall, where clients require transmission of test results*	5.5.10.7
5.13.g	*Laboratories accredited to be in compliance with these standards shall*	5.5.10.2.m
5.14	*SUBCONTRACTING ANALYTICAL SAMPLES*	5.4.5
5.14.a	*The laboratory shall advise the client in writing of its intention to*	5.4.5.2
5.14.b	*Where a laboratory subcontracts any part of the testing covered under*	5.4.5.1
5.14.c	*The laboratory shall retain records demonstrating that the above*	5.4.5.4
5.15	*OUTSIDE SUPPORT SERVICES AND SUPPLIES*	5.4.6
5.15.a	*Where the laboratory procures outside services and supplies, other*	5.4.6.2
5.15.b	*Where no independent assurance of the quality of outside support*	5.4.6.2 and 5.4.6.4
5.15.c	*The laboratory shall maintain records of all suppliers from whom it*	5.4.6.3
5.16	*COMPLAINTS*	5.4.8